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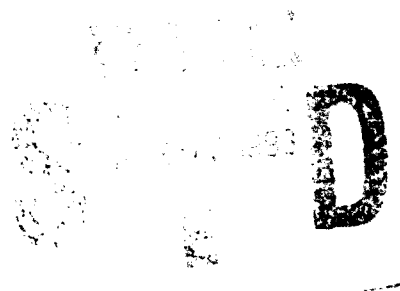
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Boron Carbide Aluminum Cermets for External Pressure Housing Applications

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ADMINISTRATIVE INFORMATION

This work was performed during FY 1991 under project RV36I21, managed by Naval Command, Control and Ocean Surveillance Center (NCCOSC) RDT&E Division under the Independent Exploratory Development (IED) Program. The work was funded under program element 0602936N and contract N66857-91-C-1034 and was performed by personnel from NCCOSC RDT&E Division, Dow Chemical Company, and Allied Technical Services.

Released under authority of
N. B. Estabrook, Head
Ocean Engineering Division

EXECUTIVE SUMMARY

OBJECTIVE

A program was initiated by the Naval Command, Control and Ocean Surveillance Center for the development of processing techniques to fabricate large B_4C/Al cylinders and hemispheres. The overall objective of this program was to fabricate and test B_4C/Al parts representative of typical pressure housing configurations. The objective of the first stage of this program was to (1) select a B_4C/Al composition and a processing methodology to obtain materials with the required combination of compressive strength, stiffness, flexure strength and toughness, (2) demonstrate the feasibility of manufacturing never before fabricated 6-inch-diameter by 9-inch-long cylinders out of B_4C/Al , and (3) and qualify those cylinders for service as deep submergence pressure housings.

RESULTS

The pressure housing tests of B_4C/Al ceramic composites showed that the compressive strength of this material in the form of 6-inch-diameter cylinders was in excess of 300 kpsi. The B_4C/Al composite monocoque cylinders with $t/D_0 = 0.0345$, $L/D_0 = 1.49$, and 0.36 weight to displacement ratio fabricated by Dow Chemical Company have been found to be acceptable for external pressure service to 9000 psi with a $SF = 1.5$ when the ends are radially supported by ceramic composite, or titanium hemispheres.

CONCLUSION

B_4C/Al composite material is an excellent choice for construction of external pressure housings in deep submergence underwater vehicles. The payload capacity of B_4C/Al housings for 9000 psi service pressure surpasses that of titanium housings with the same service pressure rating by a factor of five.

RECOMMENDATION

The development of fabrication procedures for B_4C/Al cylinders should be continued until the fabrication technology has been sufficiently developed to permit successful fabrication of cylinders and matching hemispheres with diameters in excess of 20 inches.

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1.0 INTRODUCTION

Autonomous and remotely controlled underwater vehicles require external pressure housings with low weight to displacement ratios so that the buoyancy provided by these housings can maximize the payload carrying ability of these vehicles. Plastic reinforced with a continuous graphite fiber and monolithic 94% alumina ceramic have been successfully applied to the construction of external pressure housings for 20,000 feet design depth with 0.5 weight/displacement ratio (reference 1). Both materials satisfy only the minimum requirements of the deep submergence vehicles, so a search was made for new materials which would have improved their performances.

B₄C/Al composites, having a lower density than Al₂O₃ and a higher compressive strength than reinforced plastics, seemed to be a good candidate material for this application. Since less information is available on the processing and properties of B₄C/Al composites than on Al₂O₃, a program was initiated by the Naval Command, Control, and Ocean Surveillance Center for the development of processing techniques to fabricate large B₄C/Al cylinders and hemispheres. The overall objective of this program was to fabricate and test B₄C/Al parts representative of typical pressure housing configurations. The objective of the first stage of this program was to (1) select a B₄C/Al composition and a processing methodology to obtain materials with the required combination of compressive strength, stiffness, flexure strength and toughness, (2) demonstrate the feasibility of manufacturing never before fabricated 6 inch diameter by 5 inch long and 6 inch diameter by 9 inch long cylinders out of B₄C/Al, and (3) successfully pressure test those parts in NOSC laboratories.

2.0 EXPERIMENTAL

2.1 MATERIALS

Two B₄C powders, ESK 1500 and ESK 1500 S were used in this work. ESK 1500 has a 1.34 weight % oxygen, 0.30 wt. % nitrogen and 20.71 wt. % carbon content. The main impurities are 760 ppm of Si, 420 ppm of Fe, 307 ppm of Ca and 300 ppm of Ti. The average particle size is about 3 μ m with a surface area of 8.9 m²/g. The ESK 1500 S contains 0.67 wt. % oxygen, 0.04 wt.% nitrogen and 21.27 wt. % carbon. The major impurities were 150 ppm of Si, 250 ppm of Fe and 75 ppm of Ti. The average particle size is 3.8 μ m and the surface area is 6.84 m²/g. Porous B₄C preforms were made from the two grades of ESK powder and filled with 1145 aluminum alloy that contained 0.55 wt. % Si+Fe, 0.05 wt. % Cu and 0.05 wt. % Mn.

2.2 SAMPLES PREPARATION

In order to prepare boron carbide preforms, boron carbide powders were dispersed in distilled water. The suspensions were ultrasenically agitated, pH adjusted using NH₄OH,

aged, and cast into 4 × 4 inch forms on a plaster of Paris mold. The B₄C blocks were dried and then baked for 30 minutes at temperatures from 1000°C to 1750°C or sintered above 2000°C. The baking treatment at 1000°C to 1750°C resulted in preforms with the same density, but different surface characteristics. Sintering above 2000°C resulted in boron carbide with similar surface characteristics, but with different density (ranging from 52-85 %). The infiltration of porous boron carbide blocks was conducted under 100 millitorr vacuum at 1180°C for 1 hour 45 minutes. The procedure for preparing the boron carbide cylinders is described in section 4.0.

2.3 CHARACTERIZATION AND TESTING

The area of the aluminum melting endotherm in a high temperature DSC scan was used as a measure of the reactivity between B₄C and Al at temperatures between 1000°C and 1750°C. The data was collected using a Perkin-Elmer DTA 1700 interfaced to a computer. The samples were heated in alumina crucibles at about 20°C/min under high purity argon flowing at 40 cc/min. A high purity aluminum sample (>99.999% pure) was used as a standard. The percent aluminum metal was given by $A/B \times 100$, where A is the peak area in cal/g of the Al melt endotherm in the sample and B is the same for the Al standard. The precision and accuracy of this method was about 2 percent. The applicability of DSC for determination of the free metal content was verified by performing stereology measurements on backscattered electron images of the microstructure. The results showed less than a 2% difference between methods.

Crystalline phases were identified by x-ray diffraction with a Philips diffractometer using CuK α radiation and a scan rate of 2° per minute. The chemistry of all phases was determined from electron microprobe analysis of polished cross-sections using a CAMECA CAMEBAX electron probe. The accuracy in the determination of elemental composition was better than 3% of the amount present.

The broken pieces from 4-point bend testing were used to measure the density in an Autopycnometer 1320 (Micromeritics Corp.) with an accuracy of 0.01 g/cm³.

Samples for transmission electron microscopy (TEM) were prepared in the following manner: 3 mm diameter × 1 mm thick discs, cut with an ultrasonic disc cutter, were thinned to 100 μ m with diamond grinding plates and then polished on each side with diamond to a 1 μ m finish. Dimpling with 1 μ m diamond reduced the thickness to 15 μ m. The final thin section was obtained by Ar ion/atom milling from both sides using a liquid nitrogen cooled stage, 5 keV and 2–3 milliamperere current.

The bulk hardness was measured on surfaces polished successively with 45, 30, 15, 6 and 1 μ m diamond paste and finally finished using a colloidal silica suspension on a LECO automatic polisher.

Fracture toughness was measured using the Chevron notched bend beam technique with 4 × 3 × 45 mm samples. The notch was produced with a 250 μ m wide diamond blade; the notch depth to sample height ratio was 0.42. The Chevron notched specimens

were fractured in 3-point bending using a displacement rate of 1 $\mu\text{m}/\text{minute}$. All specimens exhibited stable crack growth throughout the fracture experiment. An average of 5 to 7 measurements were reported. The fracture toughnesses were calculated using the method developed by Shang-Xian (reference 2).

The flexure strengths of the $\text{B}_4\text{C}/\text{Al}$ composites were measured in four-point bending using the ASTM method C1161 for measuring flexure strengths of advanced ceramic materials at ambient temperatures. The specimen size was $3 \times 4 \times 45 \text{ mm}$. Flexure strength was determined from an average of 8 to 10 measurements. The upper and lower span dimensions were 20 and 40 mm, respectively. The specimens were broken using a crosshead speed of 0.5 mm/min.

Elastic modulus of the $\text{B}_4\text{C}/\text{Al}$ composites was evaluated using the pulse-echo method which determined the velocity of an ultrasonic wave traveling through the material. This technique uses a single ultrasonic transducer which acts as both the transmitter and receiver of an ultrasonic pulse. Short bursts of ultrasonic energy are introduced into a test specimen ($\sim 3 \times 4 \times 20 \text{ mm}$ in size). The time for the pulse to travel through the specimen and reflect back to the transducer is measured. This measurement along with specimen density and dimensions are used to calculate the elastic modulus.

The uniaxial compressive strength of the $\text{B}_4\text{C}/\text{Al}$ materials were measured following the technique developed at the Army Materials Testing Lab (reference 3). The compressive strength specimen was placed between WC load blocks which were attached to two loading platens. The loading platens were parallel to within less than 0.0004 inch. The compressive specimens were loaded to failure using a crosshead speed of 0.02 in/min. The compressive strength was calculated by dividing the peak load at failure by the cross-sectional area of the specimen.

In order to characterize the ability of the $\text{B}_4\text{C}/\text{Al}$ materials to resist cyclic load conditions, a stepped-stress cyclic fatigue test was utilized. This test was developed as a quick screening method to rank the compressive cyclic fatigue properties of the boron carbide aluminum materials. In this test, $\text{B}_4\text{C}/\text{Al}$ specimens approximately 1/4-inch diameter by 3/4-inch long were cycled at 0.2 Hz between a minimum (σ_{\min}) and maximum (σ_{\max}) compressive stress of 15 and 150 ksi, respectively. If the specimen survived 200 cycles under this condition, σ_{\min} and σ_{\max} were increased to 20 and 200 ksi, respectively and the test continued for another 200 cycles. If the specimen survived this condition, σ_{\min} and σ_{\max} were increased a third time to 25 and 250 ksi, respectively. This loading condition was kept constant until specimen failure or a total of 600 more cycles was reached. If the specimens survived without failure after 600 cycles, the test was stopped and the specimen unloaded. The results were plotted in terms of the maximum applied compressive stress (σ_{\max}) versus the total number of cycles the specimen survived during the test.

Machined cylinders were inspected for internal defects by x-ray transmission with an IRT HOMX-161, s/n 155 equipped with a micro-focus x-ray tube (tungsten anode) operated with a five micron focal spot. The system was equipped with both a film cassette for film radiography and an IRT ADR550 system for digital image generation. The digital

images were obtained through a Precise Optics image intensifier and Videospection plum-bicon diode camera.

3.0 SELECTION OF A B_4C/Al COMPOSITION FOR PRESSURE HOUSING APPLICATION

B_4C/Al represents a rather complex family of materials ranging from cermets to ceramics. Depending on the processing methodology several B_4C/Al composites with different combinations of properties and microstructures can be produced. Therefore, to select a material optimized for the pressure housing application, the evaluation of various B_4C/Al materials was conducted. The main evaluation criteria were based on the chemistry, microstructure, properties and processability. The optimization was done mainly through adjusting boron carbide content (from 50 to 80 volume %) and through changing the boron carbide heat-treatment.

3.1 CHEMISTRY AND MICROSTRUCTURES OF THE B_4C/Al SYSTEM

The processing methodology applied in this work is based on the infiltration method where a porous boron carbide preform is infiltrated at about 1200°C with liquid aluminum (reference 4). The larger the part, the longer the infiltration time. Therefore, the issue of chemical compatibility and the recognition of the type of phases forming during infiltration becomes essential for tailoring the material properties.

The main part of the fabrication process before infiltration is the preparation of B_4C preform. The heat-treatment or baking of boron carbide is critical to control connectivity of B_4C grains, shrinkage of the preform and its chemical reactivity with molten Al. These properties change as a function of baking temperature. At baking temperatures below 1300°C, the B_4C preforms have a similar microstructure to non-treated greenware. No shrinkage in the preform is observed and the grains do not develop strong necking. The preforms consist of grains having a bimodal distribution with about 50 % large and 50 % small crystals. At higher baking temperatures, the small B_4C grains disappear as the structure coarsens and the weak necks between grains develop. Above 1600°C, shrinkage in the preform becomes noticeable, but is not significant. Above 1800°C sintering occurs and changes in the part dimensions are significant.

The surface characterization (by ESCA) of boron carbide powders baked above 1300°C has shown an increase in the total carbon content as a function of increasing temperature. This carbon is not bonded to boron and is not in the form of graphite. Since B_4C is described as B12 icosahedral clusters with C-C-C intericosahedral chains, this increase could represent a change in the chemistry of the boron carbide. The depth of the ESCA beam into the surface is only about 50Å. Further analysis of the B_4C powders using TEM (beam depth of 200 Å) has indicated that below this carbon-rich layer, the B/C ratio actually increases with bake temperature. The richer carbon surface with respect to the

deficient carbon underlayer suggests the preferential diffusion of carbon towards the surface during baking. However, the exact mechanism for the carbon-rich layer formation is unknown at this time. It is known that boron carbide has a broad solubility range for C which ranges from 9 to 21 atomic %. Therefore, even though the diffraction pattern does not change, this formation of a boron carbide surface layer with a modified chemistry is possible.

The significance of this surface change in boron carbide during baking is very important since it influences chemical compatibility of the carbide with aluminum. In greenware samples which were treated below 1300°C, the B_4C is chemically unstable with aluminum at infiltration temperatures, reacting to form several B-C-Al ternary and binary phases. Even though at least nine ternary and binary phases have been reported for the B-C-Al system only the four shown in figure 1 were found to form in significant quantities and affect properties. After infiltration is completed, the amount of residual metallic aluminum is typically below 5-6%, as shown in figure 2. As the bake temperature is increased above 1300°C, the kinetics of the reactions between B_4C and Al decreases. This transition in the reaction kinetics ends at 1600°C after which B_4C becomes chemically stable and does not react extensively with the molten aluminum during infiltration. This chemical stability is maintained even after prolonged infiltration times. The amount of B-C-Al phases formed in the materials is typically less than 10 vol.%. These observations correlate well with theoretical densities measured for B_4C /Al composites. The density for highly reacted B_4C /Al cermet (bake temperatures <1300°C) ranges from 2.66 to 2.63 g/cc. As the bake temperature increases to >1600°C, the density decreases to 2.57 g/cc as the B_4C becomes stable with molten aluminum (figure 3). The similar correlation as for baking temperature and density is observed for hardness of B_4C /Al composites. Figure 4 shows that as the amount of reaction phases in the B_4C /Al composite decreases a corresponding decrease is observed in the hardness.

The microstructural differences between B_4C /Al materials processed at different baking temperatures are illustrated on microphotographs presented in figures 5 and 6. The composites baked at and below 1300°C have boron carbide grains isolated by a matrix consisting of reaction phases of AlB_2 and Al_4BC which form large patches approaching 30 to 50 μm in size. The B_4C grains have bimodal distributions with an approximate ratio of small to large grains of about 1:1. At bake temperatures of 1600°C and 1750°C, the boron carbide forms into agglomerated pockets of sintered grains. Generally, there is an absence of small B_4C grains. These pockets, however, do not form a well developed network as observed in material baked above 1800°C. The reaction products (<10 vol.%), AlB_2 and Al_4BC are uniformly distributed throughout the aluminum matrix. Al_4C_3 , which is not detectable below bake temperatures of 1300°C, is now present in the trace amounts. The phase chemistries of B_4C /Al composites prepared from green (no bake), 1300°C, 1600°C, and 1750°C baked boron carbide preforms are shown in table 1.

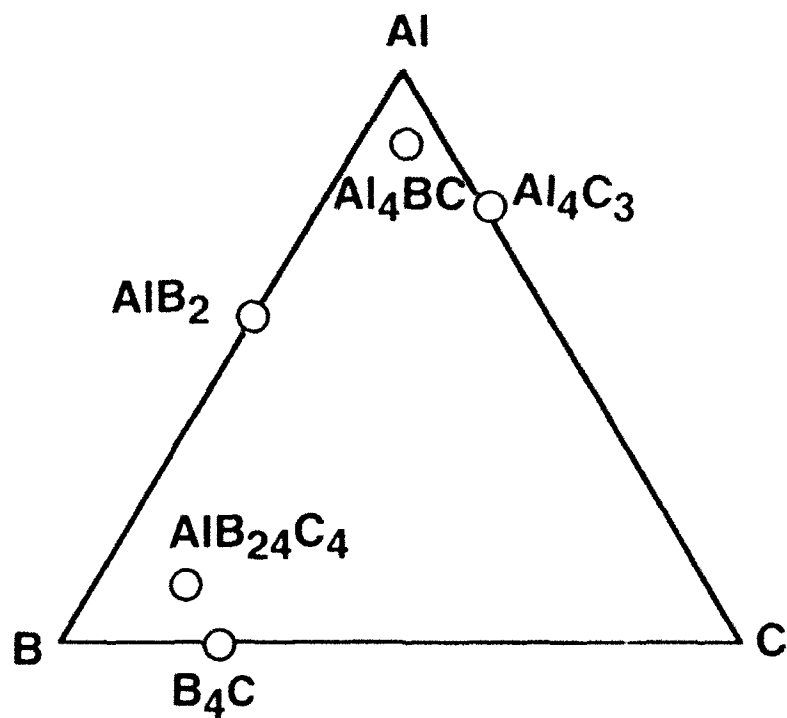


Figure 1. Major ceramic phases in the B_4C/Al system (in weight %).

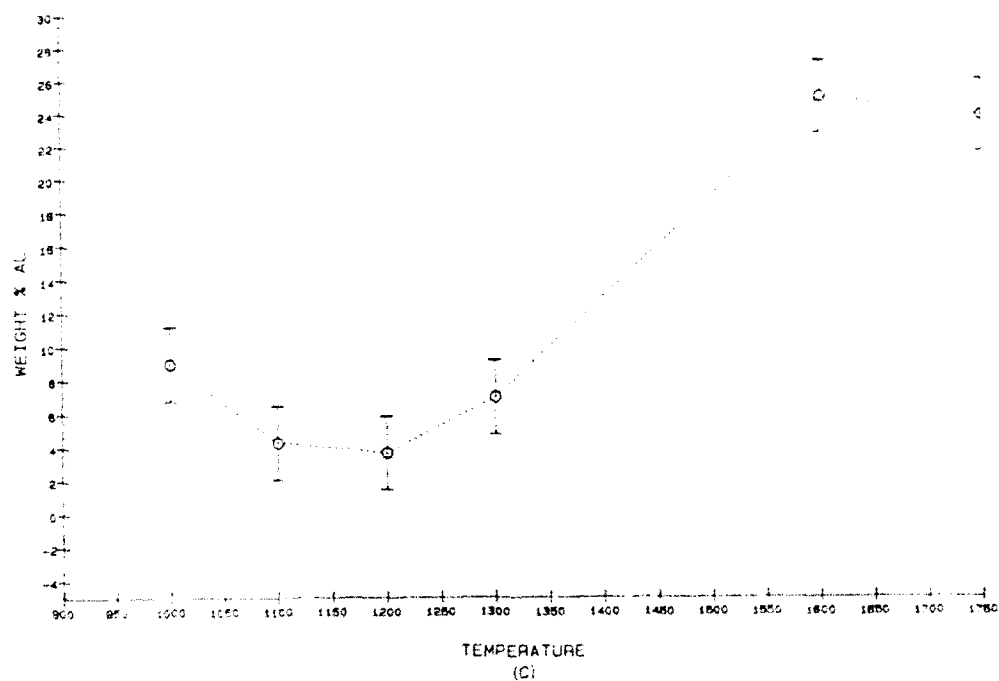


Figure 2. DSC scan of B_4C/Al prepared from boron carbide baked at different temperatures.

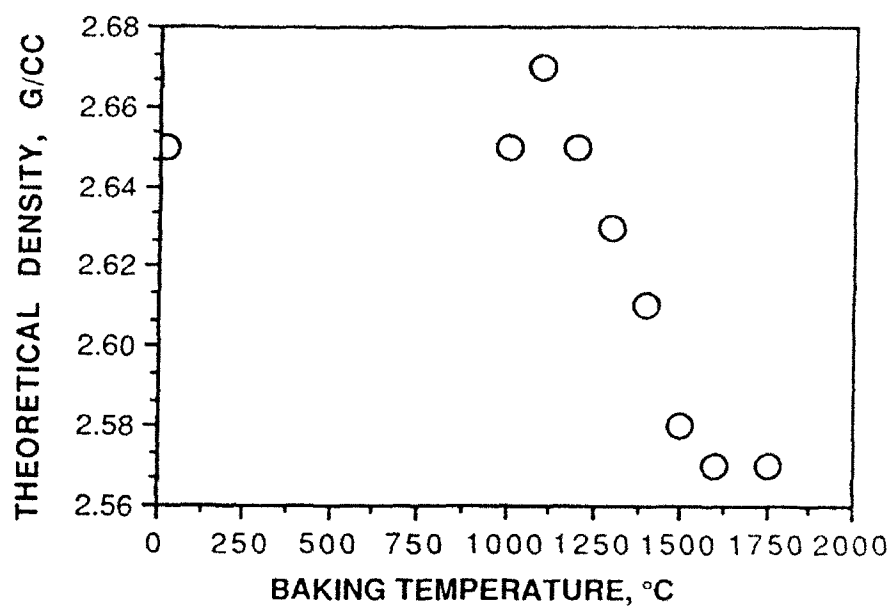


Figure 3. Theoretical density of B_4C/Al composites as a function of boron carbide bake temperature.

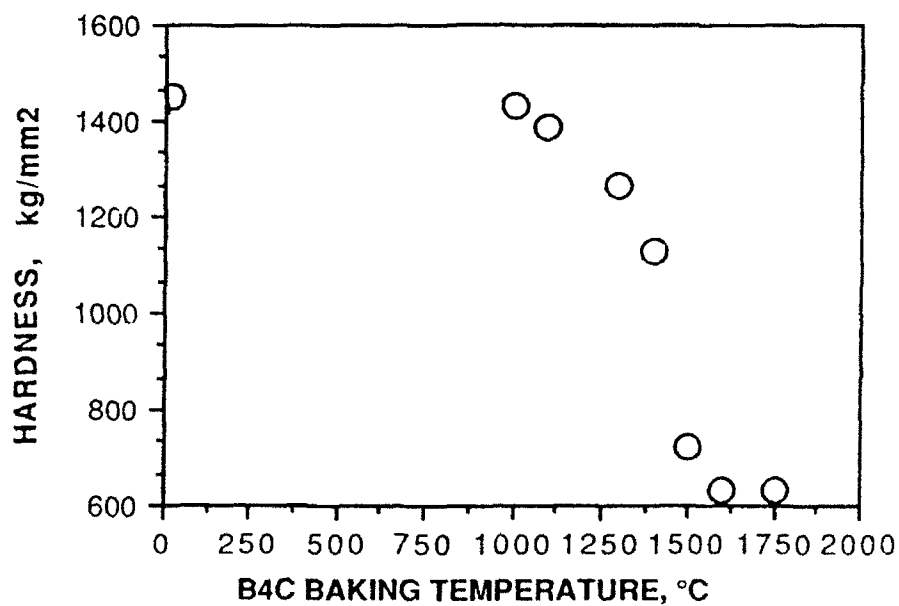
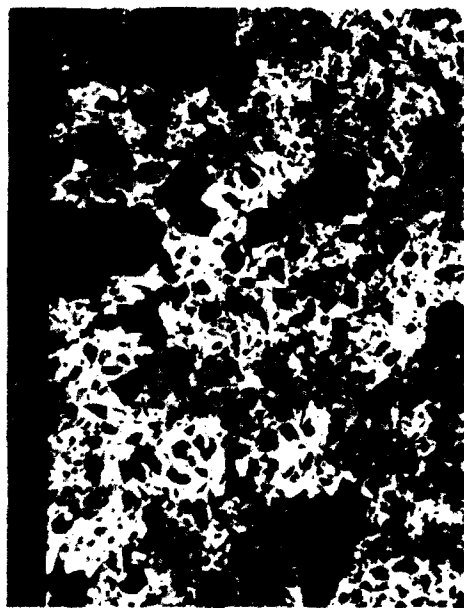
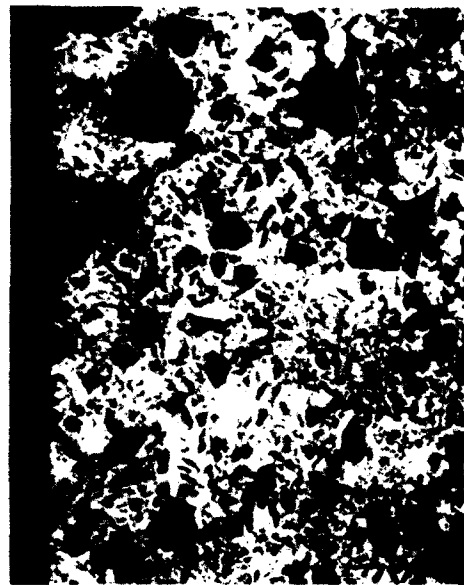


Figure 4. Hardness of B_4C/Al composites as a function of boron carbide bake temperature.



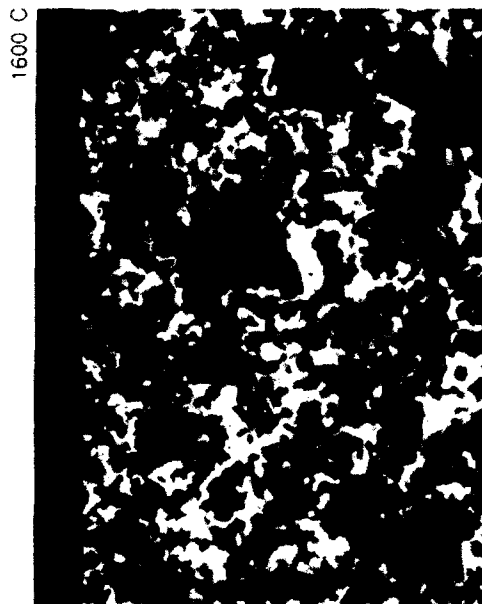
green



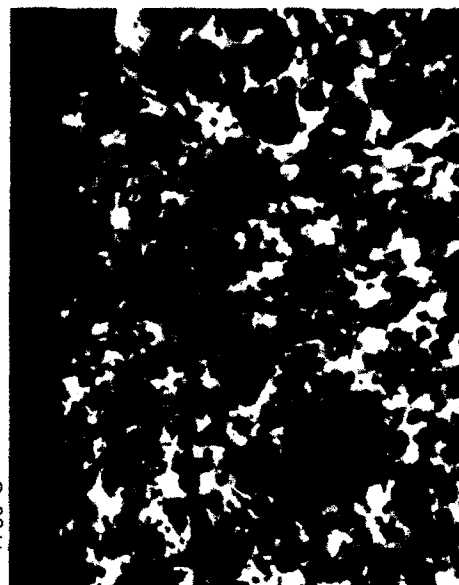
1300 C

B_4C \blacktriangle
 Al \blacktriangleleft
 AlB \blacklozenge
 Al_2BC \blacktriangleright
 Al_2O_3 \blacktriangleright
 $AlFeSi$ \blacktriangledown
 Void \sim

B_4C pre-sintered at the indicated temperature and infiltrated with 1145 Al at 1160 C for 1 h 45 min



1600 C



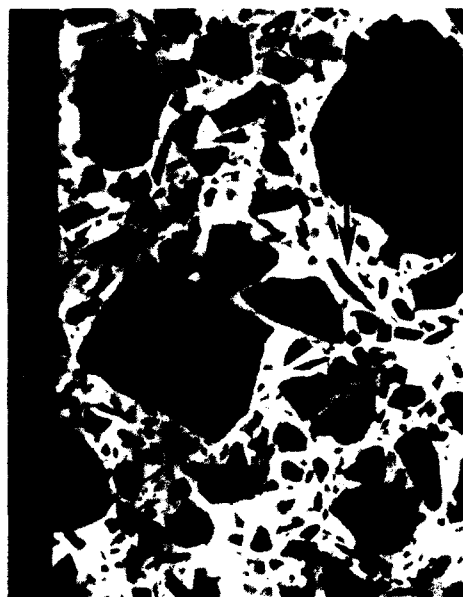
1750°C

backscattered
 electron
 micrographs
 at
 2000X
 5 μm

Figure 5. Microstructure of B_4C/Al composites fabricated from nonbaked and baked at 1300°C, 1600°C, and 1750°C boron carbide (magnification 2000x).



1300 °C



1600 °C

B_4C pre-sintered at the indicated temperature and infiltrated with 1145 Al at 1160°C for 1 h 45 min



1750 °C



1600 °C

backscattered
electron
micrographs
at
5000X
1 μ m

Figure 6. Microstructure of B_4C/Al composites fabricated from nonbaked and baked at 1300°C, 1600°C, and 1750°C boron carbide (magnification 5000 \times).

Table 1. Phase chemistry of B₄C/Al composites as a function of baking temperature (by stereology).

Composition	Baking temperature			
	20°C	1300°C	1600°C	1750°C
B ₄ C*	55.2	60.8	66.0	66.4
Al	1.8	3.6	26.9	23.9
AlB ₂	20.0	17.0	2.4	4.6
Al ₄ BC	23.0	18.6	4.7	4.1
Al ₄ C ₃	0	0	trace	1.0

*B₄C in this table represents mixture of B₄C and AlB₂₄C₄

The B₄C/Al materials processed at temperatures below the transition temperature (1300°C) are chemically similar to ceramics (little free Al metal). The B₄C/Al materials processed with boron carbide baked above this temperature have characteristics of cer-mets with boron carbide and aluminum being the primary phases. By adjusting initial B₄C to Al ratio and by changing the processing conditions, the properties of the B₄C/Al cer-mets can be tailored for specific applications. In the next section, the properties of B₄C/Al composites as a function of boron carbide content and bake temperatures are discussed.

3.2 MECHANICAL PROPERTIES OF B₄C/Al COMPOSITES

3.2.1 Compressive Strength

The effect of initial B₄C content on the compressive strengths of materials baked at different temperatures is shown in figure 7. The general trends indicate that higher initial boron carbide contents increase the overall compressive strength of the material. This result is expected since pure boron carbide has about an 800 ksi compressive strength (reference 3). Comparing the effect of bake temperature on compressive strength, the materials baked at 1750 and 2200°C had a lower strength than the green material (no bake). The lower compressive strength was attributed to the relatively high fraction of unreacted aluminum metal in these materials. Aluminum metal has a much lower compressive strength (yield strength) than boron carbide. For the green material, the compressive strength is higher because, during infiltration, the aluminum metal reacts to form non-metallic phases which have a higher compressive strength than aluminum.

Comparing the compressive strength results between the green (no bake) and the 1300°C baked materials, the compressive strength of the green material was found to be much more dependent on the initial boron carbide content. The compressive strength of the green material increased linearly from approximately 200 ksi to 650 ksi for boron

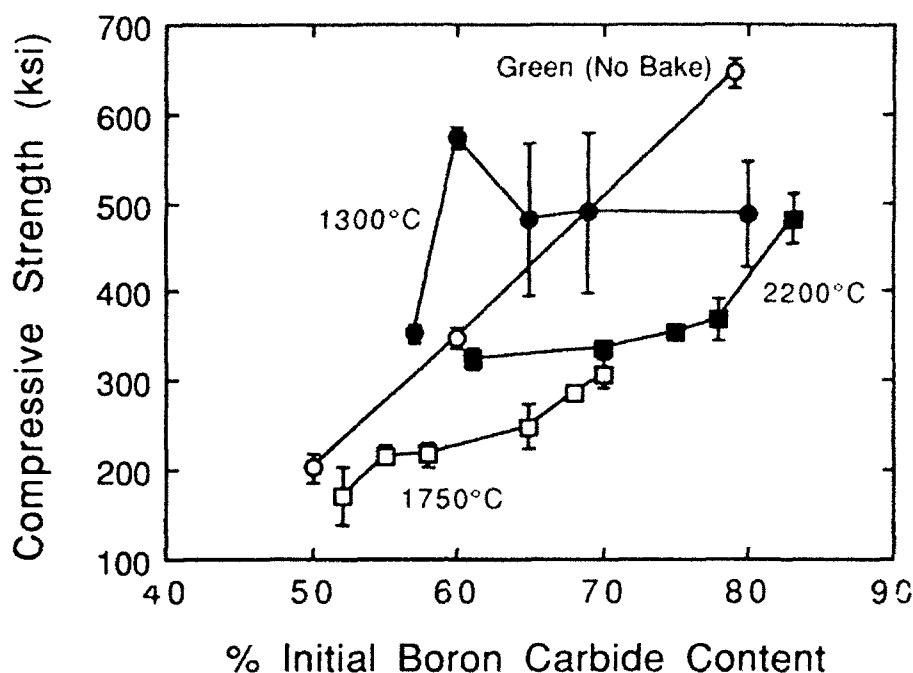


Figure 7. Effect of bake temperature and boron carbide content on B_4C/Al compressive strength.

carbide contents ranging from 50 to 78%. The 1300°C baked material, on the other hand, was found to exhibit a slightly different behavior. The compressive strength increased with boron carbide content up to approximately 70% whereupon the strength became relatively independent of the initial boron carbide content. This trend suggests that the 1300°C bake temperature makes the compressive strength of the B_4C-Al material less sensitive to initial boron carbide content. The cause of the large degree of scatter in some of the measurements is unknown at this time. To the most part, the compressive strength of the 1300°C baked material is comparable to the green material for boron carbide contents less than 70%. However, an unexpected result was observed in the 60% boron carbide material. This composite was found to exhibit compressive strengths much higher than expected. The microstructural examination has indicated the presence of large amounts of AlB_2 and Al_4BC . The microstructure of 78% boron carbide material showed an absence of Al_4BC phase which is commonly observed in this heat-treated material. Further work is required to understand the relationship between the B-C-Al intermetallic phases and compressive strength.

3.2.2 Elastic Modulus

Results for the green, 1300°C, 1750°C, and 2200°C baked materials are summarized in figure 8. As the initial B_4C content is increased, the elastic modulus for the B_4C/Al composites also increases. This result is expected because the elastic modulus of pure B_4C is approximately 450 GPa (reference 5). Comparing the different bake temperatures, it is apparent that the elastic modulus of the green and 1300°C baked materials are very

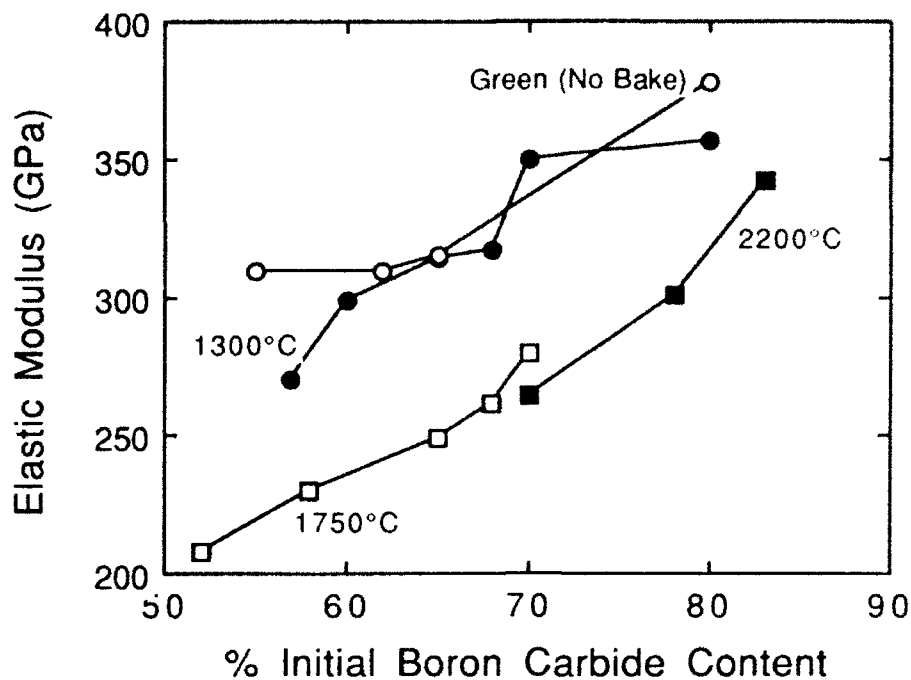


Figure 8. Effect of bake temperature and initial boron carbide content on the elastic moduli of B_4C/Al composites.

similar. The elastic moduli of these materials are also higher than the 1750°C or 2200°C baked cases. Even though the 1750°C and 2200°C materials were not evaluated over the same initial boron carbide contents, the elastic modulus results suggest that these materials have similar responses. The differences in elastic moduli in the materials bake below 1300°C and above 1750°C can be attributed to microstructure. The materials baked above 1750°C contain approximately ~23 to 24 vol.% free aluminum metal (see figure 3). In comparison, the green and 1300°C baked materials have very little free aluminum metal (~< 6 vol.%) and are comprised mainly of B_4C and non-metallic phases of B, C, and Al. These non-metallic phases have a higher elastic modulus than free aluminum metal.

3.2.3 Fracture Toughness

The results of the fracture toughness measurements are shown in figure 9. Both the green and 1300°C bake materials have very similar fracture toughnesses throughout the range of B_4C contents investigated. The fracture toughness for these materials was observed to decrease from ~7 MPa $m^{1/2}$ at ~55% initial B_4C content to a constant value of ~5 MPa $m^{1/2}$ at 80% initial B_4C content. The materials baked at 1750°C and 2200°C also show a decrease in fracture toughness with increasing B_4C content. However, the fracture toughness of these materials is slightly higher than the green or 1300°C baked material. The higher fracture toughness is attributed to the residual free aluminum metal in the composite. The residual free aluminum provides ductility which resists crack propagation and increases the fracture toughness of the material. At higher initial boron carbide

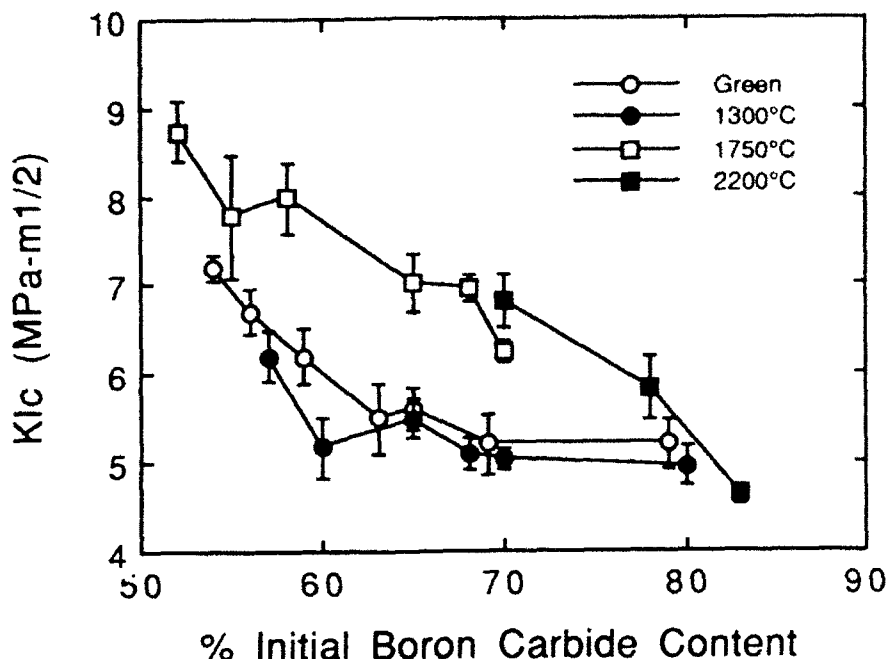


Figure 9. Effect of bake temperature and boron carbide content on B₄C/Al fracture toughness.

contents, the fracture toughness decreases due to a lower content of ductile aluminum metal.

3.2.4 Flexure Strength

The results of these measurements on green, 1300°C, 1750°C and 2200°C baked materials are shown in figures 10a and 10b. The data has been separated into two plots for clarity. The flexure strengths of the green material (figure 10a) decreased slightly with increasing boron carbide content. The 1300°C material, on the other hand, exhibited slightly more scattered strength measurements, but was found not to vary strongly with the initial boron carbide content. The reason for the large degree of scatter in the 1300°C baked material is unknown at this time. For the most part, the differences in flexure strength between the green and 1300°C material are small, particularly at initial boron carbide contents $>68\%$. The materials baked at 1750°C and 2200°C exhibited a stronger dependence on the initial boron carbide content (figure 10b). The flexure strength of the 1750°C baked material appears to increase to a maximum at initial boron carbide contents of 65–70%. The flexure strengths of this material at higher boron carbide contents were not evaluated in this study. For the 2200°C baked case, the flexure strengths of materials made with 73 to 83% boron carbide decreased with increasing boron carbide contents. It is interesting to note that the strengths of the 1750°C and 2200°C baked materials made with 70% boron carbide were very similar to each other. This result suggests that the boron carbide content plays a major role determining the strength of these materials whereas, the baked temperature only has a minor effect.

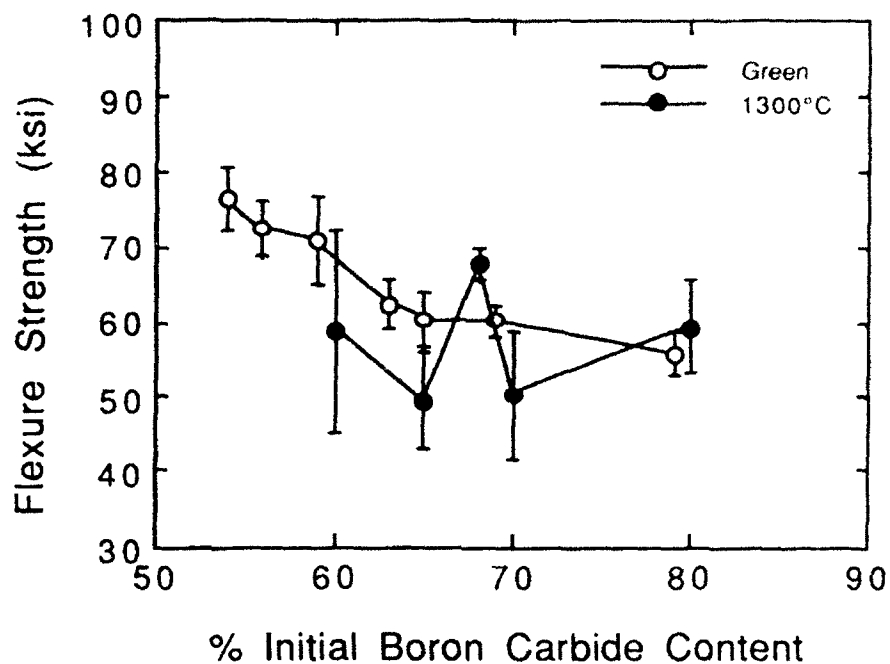


Figure 10a. Effect of bake temperature and boron carbide content on B_4C/Al flexure strength.

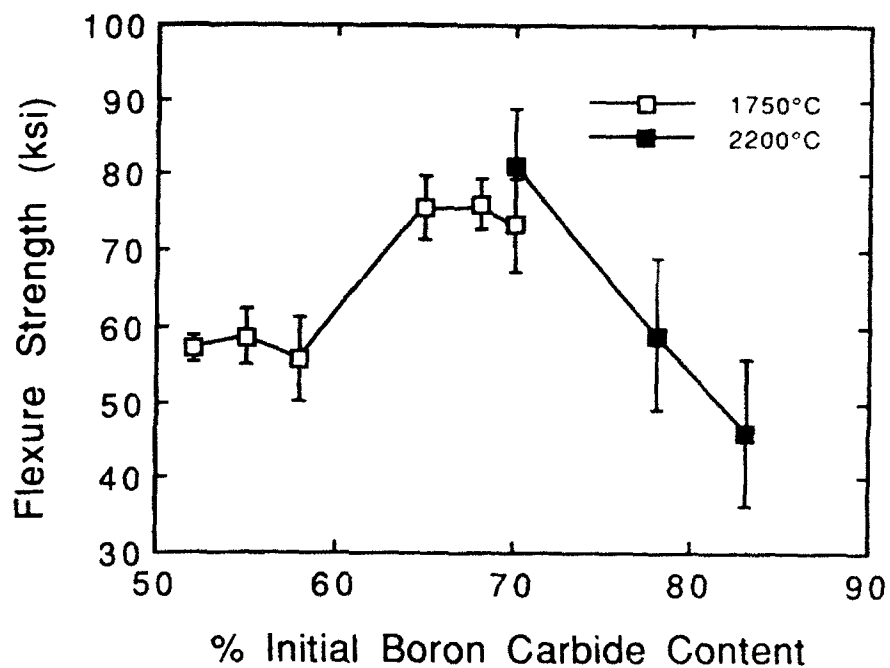


Figure 10b. Effect of bake temperature and boron carbide content on B_4C/Al flexure strength.

3.2.5 Stepped-Stress Cyclic Compression Tests

It is commonly known that cyclic loading in compression can generate damage in ceramic materials which ultimately leads to time-delayed failure at stresses lower than the compressive strength.

The stepped-stress cyclic fatigue results of the B_4C/Al materials investigated in this study are summarized in figures 11a and 11b. The results for the materials baked at 1750°C and 2200°C (figure 11a) indicate that the materials made with higher boron carbide contents have a better cyclic fatigue resistance. For example, the 1750°C baked specimens made with 52% boron carbide were only able to survive 1 and 2 cycles, respectively under a σ_{max} of 150 ksi. In comparison, the two 1750°C baked specimens made with 70% boron carbide, survived a total of 400 cycles (200 cycles at σ_{max} of 150 ksi and 200 cycles at σ_{max} of 200 ksi) before failing at -230 and 240 ksi, respectively during loading to next level of σ_{max} . A similar trend was observed in the 2200°C baked materials. The material made with 83% boron carbide exhibited the best resistance to cycle fatigue. These results also suggest that bake temperature has little effect on the cyclic fatigue response. For example, both sets of specimens baked at 1750 and 2200°C made with 70% boron carbide exhibited the same cyclic fatigue resistance. It is clear from these results that the cyclic fatigue resistance is strongly dependent on the boron carbide content in the material. This trend may be related to the fact that compressive strength of the B_4C/Al material was also found to increase with the initial boron carbide content (see figure 7).

The stepped-stress cyclic fatigue responses of the green (no baked) and the 1300°C material are shown in figure 11b. These results show a slight improvement in the cyclic fatigue resistance with increasing boron carbide content. It is unsure exactly how much better the cyclic fatigue resistance is for the 1300°C baked materials made with 70 to 80% boron carbide because the tests were stopped before specimen failure. Examining the limited data on green (no bake) materials, the cyclic fatigue resistance of green material appears to be similar to the 1300°C baked materials.

Comparing the results between figures 11a and 11b, the green and 1300°C baked materials have better cyclic fatigue resistance than the 1750 and 2200°C baked materials. These results correlate well with the trends observed in the compressive strength measurements (figure 7). The unreacted aluminum metal in the 1750 and 2200°C materials is thought to be responsible for the decrease in cyclic fatigue resistance. Figures 12a and 12b are fracture surfaces of the cyclic fatigue specimens for 1300°C and 1750°C baked materials made with 68% boron carbide. The fracture surface of the 1300°C specimen (figure 12a) is characteristic of brittle failure. On the other hand, the 1750°C baked specimens (figure 12b) contained pockets of aluminum metal that exhibited ductile failure. It is apparent that the loading capabilities of the 1750°C baked material in compression are reduced by the ductile flow of the aluminum metal.

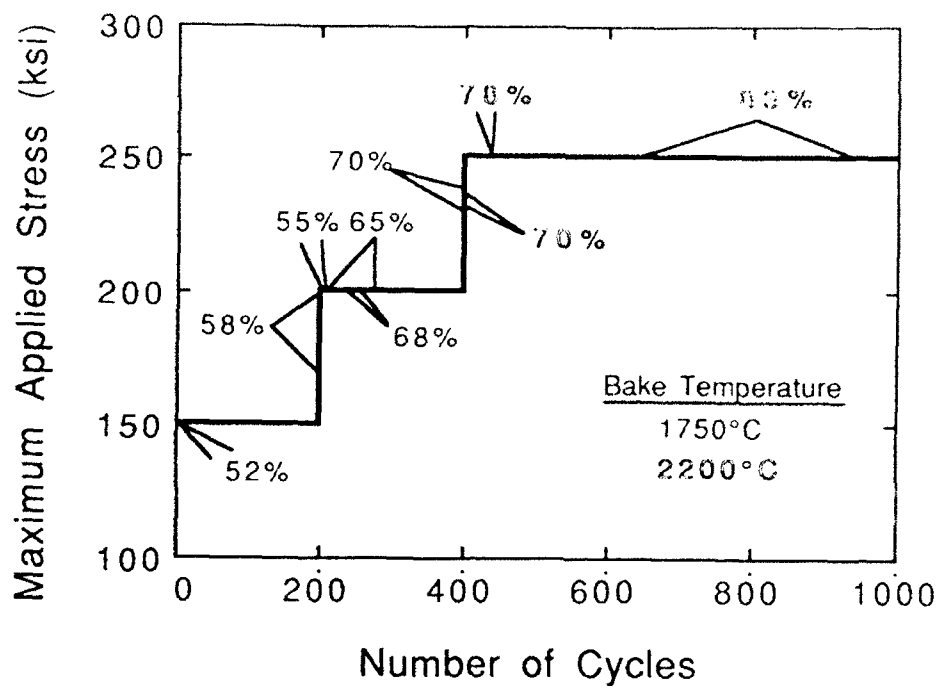


Figure 11a. Effect of bake temperature and boron carbide content on B_4C/Al cyclic fatigue.

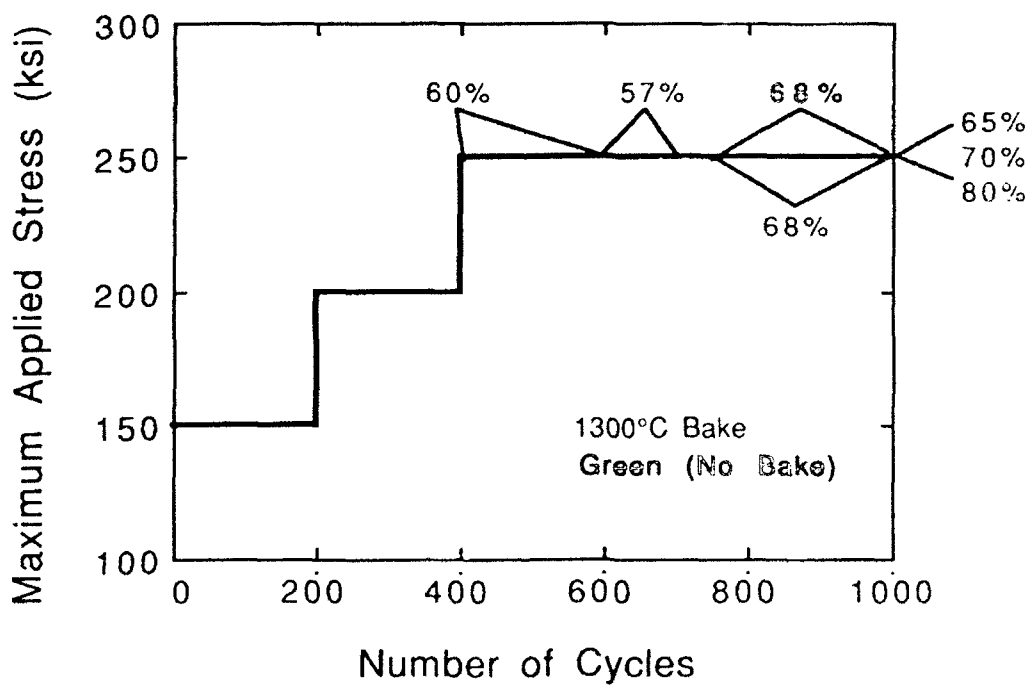


Figure 11b. Effect of bake temperature and boron carbide content on B_4C/Al cyclic fatigue.

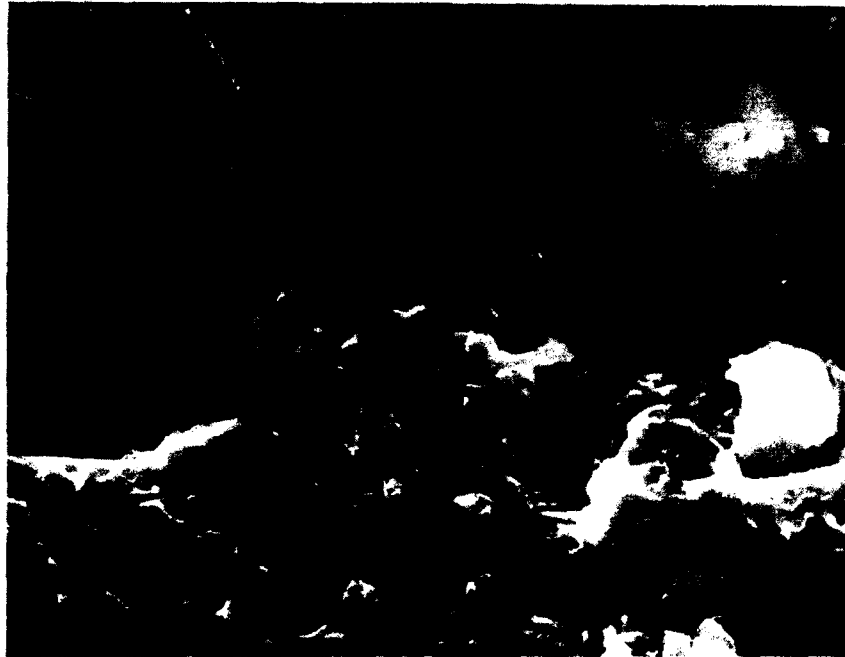


Figure 12a. Fracture surface of the 1300° baked B_4C/Al specimen.

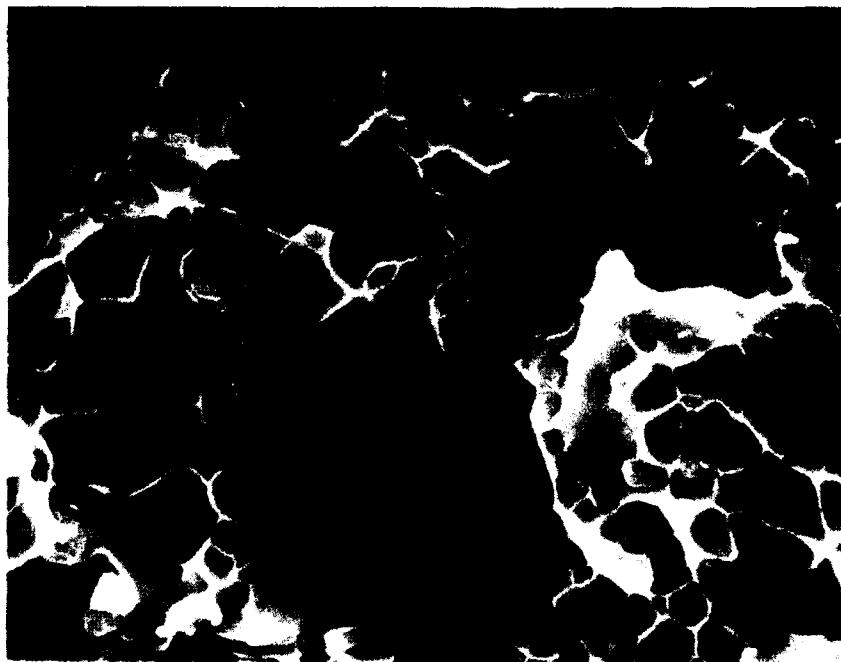


Figure 12b. Fracture surface of 1750°C baked B_4C/Al specimen.

3.3 PROCESSING CAPABILITIES

In the two previous sections we have shown that boron carbide content and heat-treatment temperature have profound effects on the chemistry and mechanical properties of B_4C/Al cermets. Since the processing of large parts represents different problems than making 4×4 inch test coupons, the processing feasibility has to be taken into account in selecting an optimum composition for pressure housing. Perhaps the best candidate material for a pressure housing is green (non-baked) boron carbide. However, making complex parts from this type of material is difficult. This is because the preform does not have sufficient structural integrity to be easily handled and the infiltration kinetics are slow. The use of a binder improves handling capability; but, after binder burn-out, the preform is fragile and susceptible to cracking during infiltration. This problem becomes significant particularly for large parts. Therefore, the baking step is beneficial. A $1300^\circ C$ baking temperature produces strong preforms with improved kinetics of liquid metal flow but with chemistry similar to composites having green boron carbide. Baking at temperatures above $1300^\circ C$ further improves preform integrity and wettability, but also changes chemistry of final B_4C/Al product in the manner described in section 3.1.

Based upon a compromise between the processibility, chemistry and mechanical properties of the boron carbide-aluminum system, the material selected for further development into pressure housings was the 68–69% B_4C preforms baked at $1300^\circ C$ (the compressive strength data show, however, that the 60% B_4C preforms baked at $1300^\circ C$ could be also a good candidate material). A summary of the mechanical properties for selected material is listed in table 2.

Table 2. Summary of the mechanical properties of the B_4C -Al material selected for further development into pressure housing applications.

Initial Boron Carbide	Content 68–69%
Compressive Strength (psi)	490
Elastic Modulus (psi)	46×10^6
Flexure Strength (psi)	$67,700 \pm 2,260$
Fracture Toughness ($MPa \cdot m^{1/2}$)	5.09 ± 0.17
Hardness (kg/mm^2)	1,400

4.0 CYLINDER FABRICATION

4.1 FORMATION OF BORON CARBIDE PREFORM

The slips (40 volume % solids) were made from a mixture of two B_4C powders, ESK 1500 (30-40 volume %) and ESK 1500 S (70-60 volume %). The powders were gradually dispersed into high purity HO while the pH was continually adjusted with NH_4OH to maintain a pH of 7.0. Maintaining proper pH is required to avoid flocculation which results in low green density. This particular mix of powders will produce greenware with densities of about 70 % of the theoretical B_4C density. The slip was stirred for 4-5 hours and its pH was adjusted as needed. The slip was then ball milled for 12 hours with B_4C media and again stirred for 3-4 hours as its pH was monitored and adjusted.

USG No. 1 pottery plaster was used to make cylindrical molds with an inner diameter slightly greater than the outer diameter of the finished part. The five inch tall pressure housings were cast using one 6 inch high mold while the 9 inch housing required the stacking of two plaster molds which were then taped together with a water resistant tape. In both cases the bottom of the molds were sealed to prevent leaking of the slip. The molds were dried in a $50^\circ C$ oven for a minimum of 24 hours before use.

The first step in the casting of the B_4C cylinders was the degassing of the slip to remove the air that was introduced by stirring and rolling. Next, the mold was filled with distilled water for 45 seconds and then emptied. The slip was slowly poured into the plaster mold, which had been previously dried in $50^\circ C$ oven for a minimum of 24 hours. Extreme care had to be maintained during pouring to minimize any turbulence that would reintroduce air into the slip. As the slip began casting, additional slip was poured into the mold to maintain the level of the casting. The slip was allowed to cast from 2-2.5 hours depending upon the desired wall thickness. At the end of the casting period, the slip was removed from the mold by a small pump.

The mold and casting were allowed to air dry until the slip remaining in the bottom of the mold had solidified. At this point, the rim of the casting was cut away from the B_4C which had cast on the cardboard bottom of the mold. The cardboard was then removed and discarded. The mold and casting were allowed to air dry until the casting was dry enough not to slump. The mold was then carefully removed from the casting. The ends of the casting were then cleaned by cutting/scraping with a surgical scalpel. The casting was placed into a low temperature drying oven for 24 hours followed by an additional low temperature vacuum treatment for 24 hours. At this point, the cylinder was ready for further processing.

The process described above was used to produce two types of cylindrical parts:

Type 3 and 3a Housing with specifications of a 6-inch diameter, 5-inch length and a wall thickness of 0.125 inch (Type 3), or 0.135 inch (Type 3a). The as-cast dimensions were 6.05-6.07 inches, 5.5-6.5 inches, and 0.25-0.30 inches,

respectively. See figure 13 for the details of the inner diameter geometry.

Type 1 Housing with specifications of 6.037-inch diameter, 9-inch length and a wall thickness of 0.208 inch. The as-cast dimensions were 6.070–6.090 inches, 9.5–10.5 inches, and 0.27–0.32 inches, respectively. See figure 14 for the details.

Density measurements of the cylinders were often difficult to obtain due to irregularities in the cylinders, especially at the ends. At least six measurements of the inner and outer diameters were taken along the length of the cylinder. Since it was critically important to obtain an accurate density figure, small rectangular parts were cast from the cylinder slips in nylon molds on plaster bases. The density figures obtained from the cylinder and from the rectangular part were averaged and used in determining the amount of aluminum required to fill the porosity in the B₄C preform.

4.2 INFILTRATION

The cylinders were placed in a low wall dish made of a refractory material and a layer of aluminum resistant material was placed around the base to confine the molten aluminum near the cylinder bottom. Pieces of high purity (1145) aluminum cut from a roll of one inch wide extruded ribbon were placed around the cylinder.

The assembly described above was placed in a high temperature furnace and three thermocouples were used to monitor the thermal environment. Two thermocouples were placed within the cylinder with one (A) located near the top of the cylinder and the second (B) located near the bottom of the cylinder. The third thermocouple was placed outside the filling assembly. Then a 10⁻⁵ torr vacuum was pulled on the furnace.

The furnace was heated slowly with the goal being to keep the temperature difference between thermocouples (A) and (B) less than 150 degrees centigrade. The furnace was heated until the lowest temperature detected by either of the internal thermocouples was slightly less than 1150°C. After a two hour hold at maximum temperature, the furnace was cooled slowly with care taken to keep the temperature difference between thermocouples (A) and (B) again within 150°C. Special care was taken around the melting point of aluminum with a two hour hold at 100 degrees above that point and 2 hour ramp to 50 degrees below that point, followed by another 2 hour hold with slow cooling to 100°C after which the furnace could be opened. The typical part after infiltration is shown in figure 15. The residual aluminum not used during the infiltration is usually found at the bottom of the cylinder normally along the inner surface, but occasionally also along the outer surface.

TYPE 3

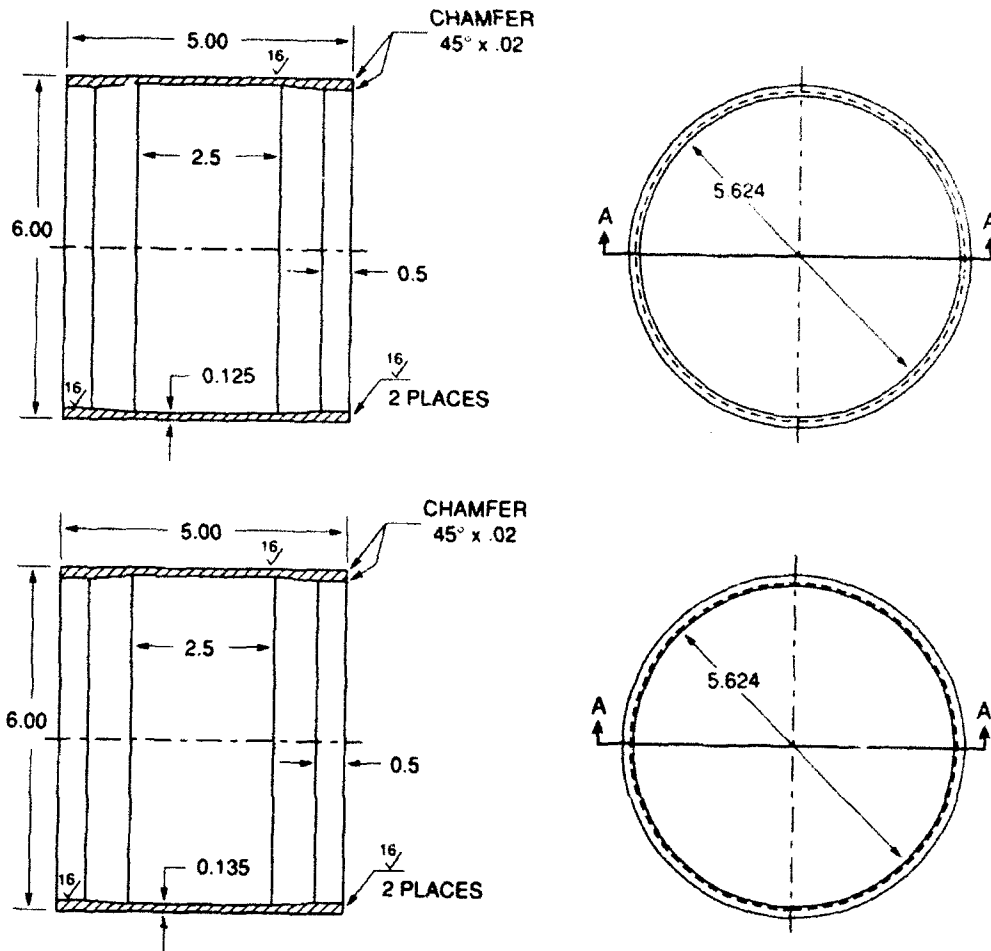


Figure 13. Specification for types 3 and 3a housings.

TYPE 1

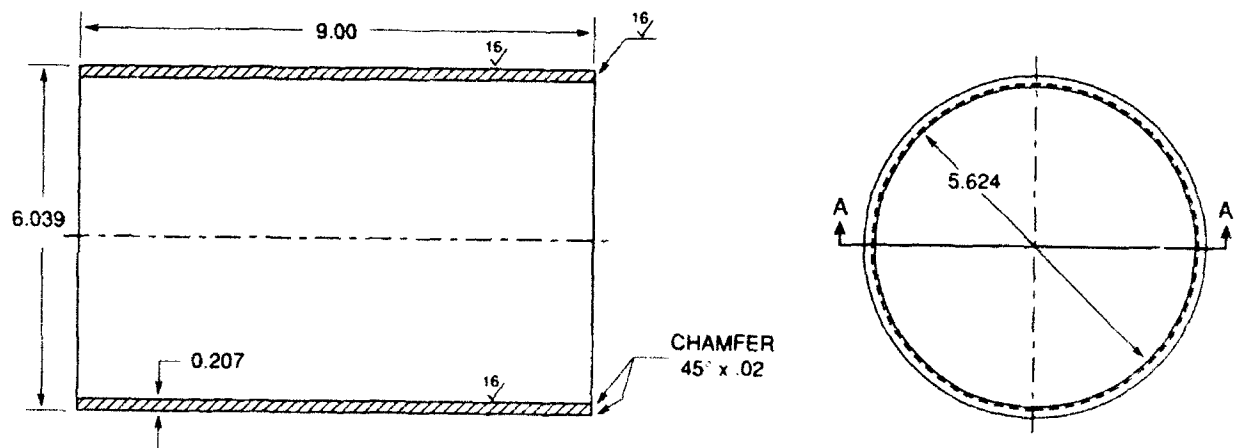


Figure 14. Specification for type 1 housing.

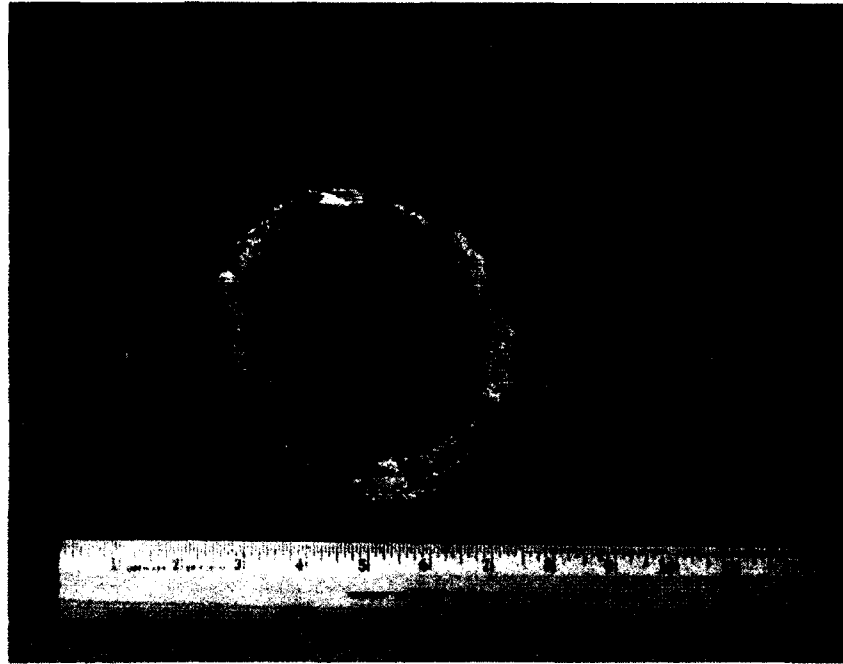


Figure 15. B₄C/Al cylinder after infiltration.

4.3 MACHINING

After infiltration, the outer surface of the cylinders are light grey in color and fairly rough in texture from the lines and scratches in the plaster molds. The inner surfaces are smooth and usually a much darker color. The ends of the cylinders were usually fairly rough from chipping of the greenware. Wall thickness reductions at the top of the casting were the result of not having enough slip to maintain the initial casting level. Type 2 and Type 3 housings were machined in a similar fashion. The first step in this process was the machining of the outer diameter. This was done by securing the cylinder in the holder pictured in figure 16a by tightening the nuts on either end just enough to hold the part so that it could be turned in a lathe. Diamond grinding wheels were used to grind away the excess stock on the outside of the cylinder. When the cylinder's outer diameter was ground to the required size, it was removed from the holder and placed in an aluminum holder (pot) as shown in figure 16b. The aluminum pot was turned on the same lathe and diamond wheels were used to grind the excess stock from the inner diameter of the housing. After this, the length was ground to the proper dimension and both the inner and outer diameters received a light grinding to the prescribed surface finish. Figure 17 shows B₄C/Al cylinders at different processing stages. The two cylinders in the front received final finish. The two in the back on left have only a rough finish while the cylinder on the right has no machining at all.

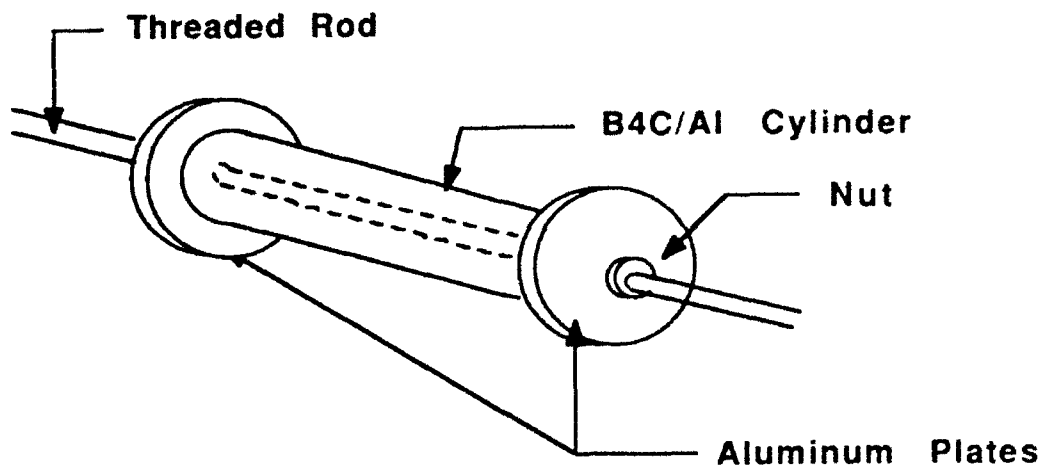


Figure 16a. Fixture for grinding the external diameter of the cast cylinder.

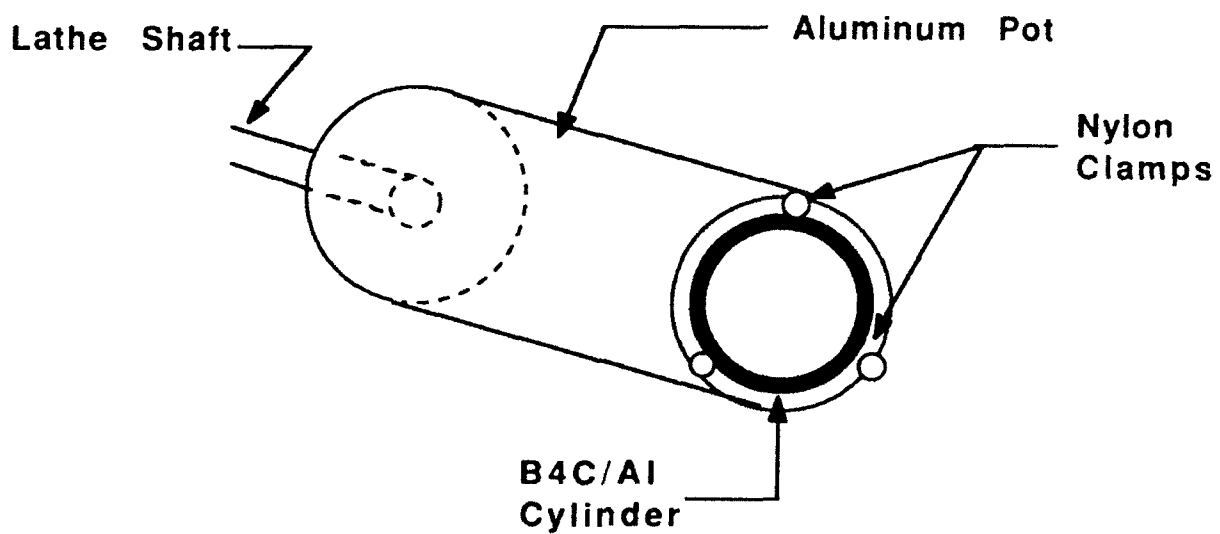


Figure 16b. Fixture for grinding the internal diameter of the cylinder after its external diameter has been already ground to net dimension.

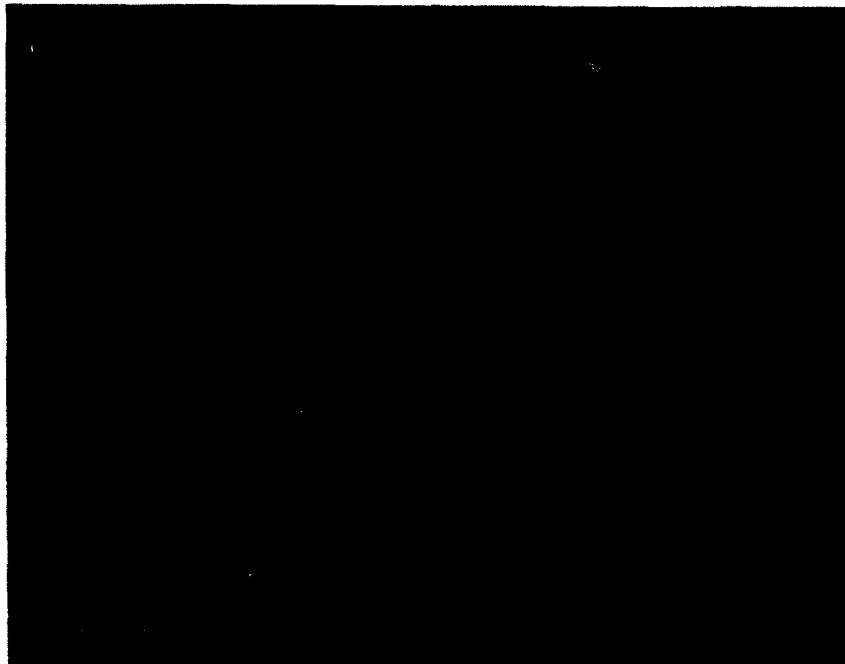


Figure 17. Some of B₄C/Al cylinders fabricated at Advanced Ceramics Laboratory of Dow Chemical Company during first stage of NOSC contract.

4.4 NDE CHARACTERIZATION

Each cylinder was inspected by mounting the cylinder on spacer blocks which held the part away from the sample positioning stage. The blocks contacted only the cylinder edge and did not intrude inside the cylinder walls. The distance from the center of the x-ray tube (focal spot) to the cylinder wall (outside diameter) was 544 mm. When x-ray film was used as a detector, the distance from the center of the x-ray tube to the film was 549 mm. If digital images were generated, a 512 × 480 pixel (8 bit) image was obtained through the IRT ADR550 frame buffering system. In the digital image arrangement, the cylinder was located at the same position as the film test, but the x-ray tube to detector distance was increased to 1076 mm. Also, the x-ray beam passed through both walls of the cylinder before detection and incorporated the additional absorption of the second wall and two different magnifications super-imposed for both-wall transmission.

During testing, the cylinders were rotated at 0.5 rpm and the irradiation conditions were 60kV, 0.5mA for a total of 30 minutes with an x-ray beam collimator. This collimator consisted of a slit 1.0 mm wide, 15 mm in length, 1.4 mm thick, mounted on the end of a tube (detector side) with a length of 13 mm and a diameter of 12 mm. The slit and tube were mounted to the x-ray tube at a distance of 18 mm in front of the x-ray tube focal spot. This collimator arrangement produced a 20 mm wide beam at the cylinder and exceeded the dimensions of the cylinders in the vertical direction.

The theory behind the x-ray transmission testing is that the strength of the transmitted signal through a piece with a uniform wall thickness is dependent upon the amount of material passed through and thus is indicative of the relative densities of adjoining sections of the cylinders. The uniformity of the B_4C/Al distribution is also detectable by the fact that the x-ray absorption levels of B_4C and Al are quite different. The light areas on the photographs are areas that did not transmit as much of the x-ray beam and conversely the dark areas are those sections that transmitted more of the beam.

No internal voids were detected in any of the cylinders submitted for NDE characterization in this study. The cylinders sent to NOSC were also determined to be free of any aluminum filled cracks. This type of defect was found to be easily identified with this x-ray inspection method.

5.0 CYLINDER PRESSURE TESTING

Ten cylinders prepared during the course of this project were subjected to external pressure testing in NRaD Laboratories. All cylinders were instrumented with electric resistance strain gauges CEA-06-125WT-350 (Micromeritics Inc.) and an acoustic emission transducer using established procedures (6,7). The ends of the cylinders were enclosed with titanium joint rings bonded to the B_4C/Al surface with epoxy resin. Testing was performed in a pressure vessel having electrical feed through which allowed the strain and acoustic emission signals to be externally monitored. For some of the testing the ends of the cylinders were radially supported by titanium hemispheres, and for other with plane steel bulkheads. Since the elastic stability of the tested cylinders depends to a large degree on the radial rigidity of the bulkheads the comparison of implosion pressures must be made only between cylinders supported by identical bulkheads. For short term pressure testing the pressures were increased in 1000 psi increments until implosion took place. During pressure cycling tests, the pressure was increased by 1000 psi/minute rate, followed by decrease at 10,000 psi/minute rate. In some cases the pressurization was terminated prior to implosion upon the first indication of non-linearity in radial displacements as experience has shown that in such a case catastrophic implosion would follow in approximately 300 psi pressure increase.

The sample characteristics and test results are shown in table 3. The purpose of the first five experiments with Type 3 cylinder housing was to determine the optimum B_4C/Al composition and green body baking temperature for pressure housing applications. The B_4C/Al cylinders tested were fabricated from 69% dense boron carbide preforms baked at 1300°C, 1400°C and 1750°C for 30 minutes and a 75% dense preform baked at 1300°C for 30 minutes (see table 3). The first cylinder (cylinder #1 baked at 1400°C) imploded at 10,500 psi pressure or a compressive hoop stress of 252,000 psi. The two next cylinders (cylinder #2 and #3) which were baked at 1300°C showed a significant improvement over cylinder 1. The implosion pressures were 12,600 psi and 11,500 psi which correspond to maximum compressive hoop stresses of 302,400 psi and 276,000 psi, respectively. The fourth cylinder was prepared from B_4C baked at 1750°C. The cylinder was initially pressurized to a design pressure of 9000 psi and then pressurized to implosion. Failure for

Table 3. Summary of pressure testing program for B₄C/Al composite cylinders.

Cylinder No.	Type	Thickness Midbay in.	Overall Length in.	B ₄ C Content vol%	Baking Temp. °C	Annealing Temp. °C	Cycling History kpsi	Maximum Hoop Strain at 10 kpsi	End Supports	Implosion Pressure kpsi	Type of Failure	Maximum Compressive Hoop Stress kpsi
1	3	.125	5	70	1400	570	1 to 10	6205	Hemis	10.5	Buckling	252
2	3	.125	5	70	1300	570	0	4837	Hemis	12.6	Buckling	302
3	3	.125	5	70	1300	570	0	4863	Hemis	11.5	Buckling	276
4	3	.125	5	70	1750	570	1 to 9	5290	Hemis	11.8	Buckling	283
5	3	.125	5	75	1300	570	1 to 12.5	4468	Hemis	12.5	Buckling#	300
5	3	.125	5	75	1300	570	2 to 12.5	4445	Plates	15.6	Buckling	374
6	3	.125	5	70	1300	0	1 to 0	4727	Hemis	13.3	Buckling#	319
6	3	.125	5	70	1300	0	1 to 13.3	4804	Plates	15.5	Buckling	372
7	1	.208	9	70	1300	570	10 to 10	2961	Hemis	10.0	Proof test	145
7	1	.208	9	70	1300	570	3000 to 9	2981	Plates	19.8	Buckling#	287
8	3a	.135	5	70	1300	570	0	4713	Hemis	13.5	Buckling	295
9	3a	.137	5	70	1300	570	0	4500	Plates	16.0	Buckling#	345
10	1	.208	9	70	1300	570	10 to 10	3073	Plates	19.5	Buckling#	285
10	1	.208	9	70	1300	570	1 to 19.5	3109	Hemis	15.6	Buckling	230

NOTES:

1. Type 1 Housing is a 6.037 in OD \times 9.0 in L \times 0.208 in thick monocoque cylinder; Wt = 1461 grams; Weight/Displacement = 0.338 without end rings, and 0.36 with end rings; PN 55910-0126847.
2. Type 3 housing is a 6.00 in OD \times 5.0 in L \times 0.125 in thick monocoque cylinder; Wt = 592 grams; Weight/Displacement = 0.25 without end rings, and 0.30 with end rings; PN 55910-0126849.
3. Type 3a housing is a 6.00 in OD \times 5.0 in L \times 0.135 in thick monocoque cylinder; Wt = 601 grams; Weight/Displacement = 0.254 without end rings, and 0.304 with end rings; PN 55910-0126849 Rev A.
4. #Testing was terminated at the onset of buckling, pressure approximately 200 to 300 psi below catastrophic implosion. These cylinders were subsequently retested with a different type of end support.

this cylinder occurred at 11,800 psi or a maximum compressive hoop stress of 283,000 psi. The testing of the fifth cylinder with a slightly higher B₄C content (75% versus 69%) and baking at 1300°C was terminated at 12,500 psi without implosion at the very threshold of failure. The highest implosion pressure, 13,300 psi, was attained with sixth cylinder which was not annealed after grinding to final dimensions; the maximum compressive hoop stress attained at 13,300 psi was 319,200.

Since there was a question whether the implosion of Type 1 cylinders was triggered by material failure, or by buckling, cylinder #5 was retested with plane steel bulkheads that provided stiffer radial support to the cylinder ends. If cylinder #5 withstood now significantly higher pressure when supported by the rigid steel end supports it would indicate that the implosions of all Type 3 cylinders in this program supported by the less rigid titanium hemispheres was triggered by buckling and not material failure. Cylinder #5 when retested with rigid steel end supports imploded at 15,600 psi, generating a maximum compressive hoop stress of 374,400 psi. The higher critical pressure achieved with rigid plane steel bulkheads serves as proof that the critical pressures and maximum hoop compressive stresses recorded during testing of Type 3 cylinders do not represent failure of material but elastic instability of the cylinders.

Two additional cylinders Type 3a #8 and 9 were provided with an 8 percent thicker shell. The reason for the slight increase in wall thickness was to raise the critical pressure of Type 3 cylinders to 13,500 psi representing the minimum acceptable safety margin for 9000 psi design pressure for cylinders supported by titanium hemispheres, similar to the bulkheads used on operational pressure housings.

The objective for testing the Type 1 cylinders #7 and 10 was to make comparison between structural performance of B₄C/Al cermet cylindrical housing and of previously fabricated 94 percent alumina with identical dimensions, and tested with identical end closures (reference 6). For this purpose, one of the Type 1 cylinders was to be pressure cycled ten times to 10,000 psi prior to implosion, while the other one was to be pressure cycled 10 times to 10,000 psi and 3,000 times to 9,000 psi. prior to testing to destruction.

Cylinder #7 withstood without failure 19,800 psi at maximum compressive hoop stress of 287,100 psi after first being pressure cycled ten times to 10,000 psi followed by 3,000 cycles to 9,000 psi. The test was terminated at 19,800 psi at initiation of non linear radial displacement. The predicted implosion by buckling was extrapolated from strain data to occur at 20,000 psi. Cylinder #10 withstood successfully 10 cycles to 10,000 psi, prior to testing to implosion which was terminated without failure at 19,500 psi. The implosion by buckling was extrapolated from strain data to occur at 19,700 psi.

Cylinder #10 was subsequently pressure tested with hemispherical instead of plane steel bulkheads to establish the critical pressure of Type 1 cylinder equipped with titanium hemispherical bulkheads typical of operational external pressure housings. The critical pressure of Type 1 monocoque cylinder was calculated by the BOSOR 4 elastic instability program to occur at approximately 20 percent lower pressure than when the cylinder ends are radically supported by infinitely rigid plane steel bulkheads.

6.0 TEST RESULTS

The results show that boron carbide heat-treatment procedure is important variable in preparing B₄C/Al cylinders for pressure housing applications. The highest implosion resistance for Type 3 cylinders was obtained with the 1300°C baked boron carbide (cylinder #2). The variations between cylinders #2 and 3 suggest possible temperature differences during the baking process. Cylinder #1, on the other hand, had the lowest implosion resistance. The lower implosion strength of cylinder #1 is thought to be related to its microstructure. At a 1400°C bake temperature, the boron carbide grains are isolated in continuous metal matrix. This continuous metal matrix cannot support compressive loads as well as the continuous ceramic matrix found in cylinder #2. Cylinder #4 which was baked at 1750°C, also performed better than cylinder #1. Even though this material contained more free Al metal, the microstructure contained a very strong continuous network of boron carbide grains. This type of microstructure resulted in a better combination of compressive strength and elastic modulus which improved the implosion pressure. Based upon these results, it appears that either the 1300°C or the 1750°C bake temperature can be used to produce cylinders with high implosion strengths. However, a bake temperature of 1750°C is less desirable as it results not only in a somewhat lower compressive strength and modulus of elasticity but also in significant part shrinkage which complicates processing since part dimensions must be controlled.

Test results also show that absence of annealing did not appear to decrease the implosion pressure; as a matter of fact, a case could be made that the absence of annealing tends to increase it. This is based on the observation that cylinder #6 with 69% boron carbide, baked at 1300°C, but annealed after grinding to final dimensions, failed at 13,300 psi, while cylinders #2 and 3 with the same B₄C/Al content baked at 1300°C, and annealed at 570°C failed at pressures of 12,600 psi and 11,500 psi respectively.

The effect of baking temperature can also be seen on the magnitude of strains recorded on the interior surface of the Type 3 cylinders at 10,000 psi proof pressure. While the average hoop strain at midbay of cylinders #2, 3 and 6 with 69% content of boron carbide baked at 1300°C was -4809 micro inches/inch it increased to -5290 for cylinder #4 baked at 1750°C, and -6205 for cylinder #1 baked at 1400°C. From this can be deducted that the modulus of elasticity reaches minimum when the baking temperature is 1400°C. Since the onset of buckling is directly related to the modulus of elasticity, the higher modulus resulting from 1300°C baking temperature is a distinct advantage, as it provides a higher safety factor against buckling. Based upon this fact, 1300°C was selected as the preferred bake temperature for the green bodies of pressure housings.

Although the 1300°C baked materials with 69-70% content of boron carbide satisfied requirements of pressure housing applications, their performance should be improved by increasing the boron carbide content in the preform. However, this is not necessarily the case for this cylinder design. Cylinder #5, which contained 75% boron carbide, did not show an improvement in implosion pressure. Since the calculated compressive strength of cylinders #2 and #5 exceeds significantly implosion pressure, it is believed that a failure mode is not entirely controlled by the compressive strength of the material. During

testing, a few acoustic emissions from the cylinders were recorded at pressures greater than 9,000 psi. This acoustic emission indicates that some deformation and/or displacement of crystals in the B_4C/Al body occurs during compression. It is thought that these displacements could be related to buckling and the subsequent collapse of the cylinder under pressure. This failure mechanism is controlled by the elastic modulus of the material and not from a lack of sufficient compressive strength. This type of failure mechanism can be eliminated by increasing the wall thickness of the pressure housing. This change in design would increase the stiffness of the cylinder, allowing the full compressive strength of the B_4C/Al material to be utilized. One drawback for increasing the wall thickness is the increase in the overall weight of the cylinder. This fact is not viewed as a disadvantage for the B_4C/Al system since this material has a very low density. For example, in comparison to the current best ceramic material for pressure housings (alumina), the density of B_4C/Al is approximately 33% less than alumina (2.63 vs 3.98 g/cc).

One interesting observation made during pressure testing of B_4C/Al cylinders was that some permanent deformation takes place during the first pressurization. Thus during the first pressurization to 10,000 psi of cylinder #7 the residual strains were on the average 100 micro inches, or 0.001 % indicating that the material has permanently deformed. During the second through 10th pressurization to 10,000 psi, no further deformation was observed. Even when this cylinder was pressure cycled once to 19,800 psi, no further permanent deformation took place.

7.0 CONCLUSIONS

1. B_4C/Al composites having a higher specific compressive strength than currently used materials are excellent candidates for external pressure housings for deep submergence autonomous underwater vehicles. B_4C/Al materials optimized for this application have chemistry and properties similar to those of ceramics, rather than to cermets. They are characterized by a compressive strength of 490 ksi, elastic modulus of 46.4×10^6 psi, flexure strength of about 55-60 ksi, fracture toughness of $5.2 \text{ MPa m}^{1/2}$ and hardness of 1400 kg/mm².
2. A procedure has been developed in The Dow Chemical Company Advanced Ceramics Laboratory that allows for the near net shape production of B_4C/Al cylinders with a diameter of 6 inch and lengths of 5 and 9 inches. The method is based on vacuum infiltration of near-net shape preforms of boron carbide which have received a proprietary baking.
3. The compressive tests performed on 6 inch diameter x 5 inch long cylinders with a wall thickness of 0.135 inch and 0.3 weight/displacement ratio showed compressive strengths of about 300 ksi, at pressures which correspond to about 30 thousand feet submergence. Since the compressive strength of B_4C/Al ceramics is higher than the compressive stress in cylinders at implosion the failure mechanism is thought to occur through buckling and the subsequent collapse of the cylinder walls. A change in cylinder design to increase stiffness would take full advantage of B_4C/Al high compressive

strength. A change to cylinder design that would increase cylinder stiffness without addition of weight is the incorporation of B₄C/Al ring stiffeners into the shell of the cylinder; such type of design has been already experimentally validated on 12-inch diameter cylinders of glass ceramic material (reference 9).

4. The B₄C/Al composite monoque cylinders with $t/D_o = 0.0345$, $L/D_o = 1.49$ and 0.36 weight to displacement ratio have been found to be acceptable (i.e., $SF > 1.5$) for external pressure service to (a) 9000 psi (20,000 ft submergence) when the ends are radially supported by ceramic composite, or titanium hemispheres, or (b) 13,300 psi (30,000 ft submergence) when radially supported by plane titanium plates. In either case, the failure mode is by buckling, and not by material failure. The weight/displacement ratio of such cylinders capped with titanium hemispheres for 9,000 psi service, is 0.48. The weight/displacement ratio can be further decreased to 0.38 if ceramic composite is substituted for titanium in the hemispheres.
5. Based on the performance of material test specimens and cylinders under cyclic compressive loading, it appears that at 150,000 psi compressive hoop stress the cyclic fatigue life of B₄C/Al composite housings is in excess of 1000 cycles.

8.0 ACKNOWLEDGEMENTS

The work was performed under U. S. Navy Contract N66857-91-C-1034. The authors express their sincere thanks to the following people for their assistance in completing this study: J. Ott, H. Rossow, S. Dorscha, J. Pretzer, J. Tokie and C. Mericle for help in fabricating cylinders and testing specimens; D. Beaman, H. Klassen, G. Mitchell, C. Wood and D. Susnitzky for microstructural and surface characterization; P. Himes for NDE evaluation; C. Black for DSC characterization; and A. Hart for support and encouragement.

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**APPENDIX A: BORON CARBIDE CERMETS
FOR PRESSURE HOUSINGS—PROCESS OVERVIEW**

Art Prunier
Dow Chemical Company

OUTLINE

- I. Preparation of Powders
- II. Preparation of Greenware Cylinder
- III. Conversion to B₄C Cermet
- IV. Machining to Size

POWDER PREPARATION

Starting Powders are from ESK: 1500 & Bimodal

Powders are washed to remove boric oxide

Methanol slurry forms trimethylborate

Centrifuge to separate powder

Dry at 50°C / trimethylborate evaporates

Washed powders are blended

69% Bimodal / 31% 1500 is ideal

Yields greenware having 31-32% porosity

Blending done in ultrapure H₂O

pH raised to 7.0 using NH₄OH

Blended powder "slip" is mixed & dispersed

Small volumes using ultrasonic treatment

Large volumes using roller mill & B₄C balls

Vacuum degassing to remove air bubbles required

PREPARATION OF GREENWARE CYLINDERS

Overall process is called "Slip Casting"

$B_4C + H_2O$ is the slip

Casting done in a Plaster of Paris mold

Mold Production

Steel cylinder machined to target size +

Plaster mold formed around steel

Steel cylinder removed, leaving cylinder -
shaped cavity of proper size

Dry mold at $50^{\circ}C$

Pour B_4C powder slurry into mold

Water pulled into porous mold

B_4C is deposited on mold walls

1.75 hr to achieve > 0.30 inch casting

Pump out excess slip

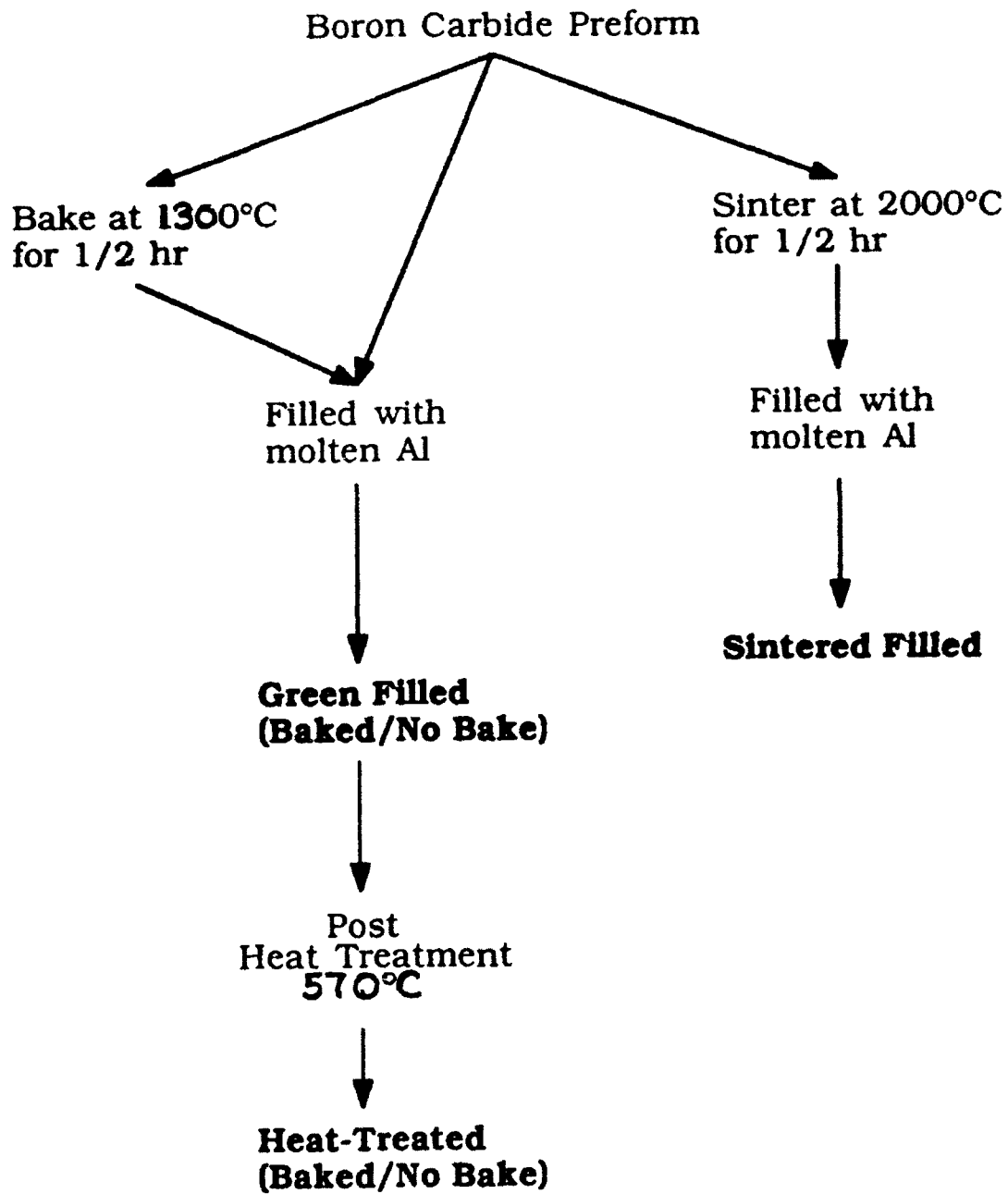
Remove cast B_4C cylinder

Dry mold + cylinder at $50^{\circ}C$

B_4C cylinder shrinks slightly, allowing it to be
removed from the mold

Dry free cylinder (= greenware) for 24 - 48 hr
at $50^{\circ}C$

Dry cylinder in vacuum oven >12 hr at $95^{\circ}C$



CONVERSION TO B₄C CERMET

Bake green cylinder, *if required*

Graphite vacuum furnace used

Prepare aluminum metal

Strips of 1145 alloy used

Calculate Al needed to fill cylinder pores

Add 7.5% extra

Prepare cylinder for infiltration

Alumina retainer used to hold parts

Distribute Al strips around cylinder

Saffil® fiber blanket holds Al next to cylinder

Actual Infiltration

At 1150 - 1180°C in vacuum furnace

Mo or W furnace preferred over C furnace

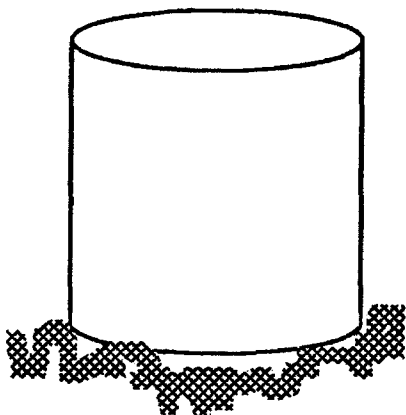
Molten Al drawn into porous cylinder

Metal can rise about 6 inches max.

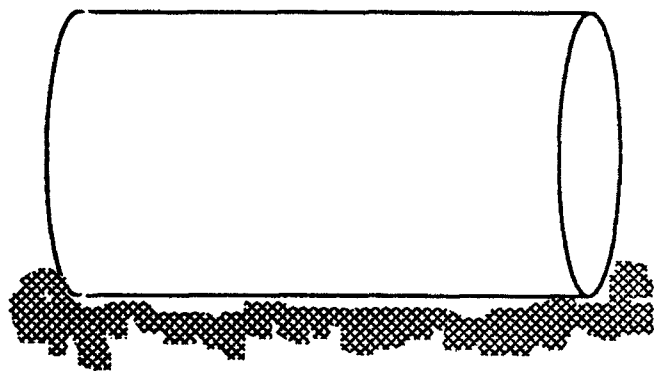
Controlled, slow cooldown

METHOD OF INFILTRATION

6 inch diameter cylinders



5 inch tall



9 inch tall

MACHINING TO SIZE

Diamond grinding must be used to achieve size specifications

Both inside and outside must be machined

Identification of competent machinist is critical

ARP / 9-91

**APPENDIX B: EXPERIMENTAL EVALUATION OF
MODEL SCALE B₄C/Al CERMET CYLINDERS
UNDER EXTERNAL PRESSURE**

J. D. Stachiw
NRaD
Code 9402

BACKGROUND

Characterization of the B₄C/Aluminum composite by testing of compression and flexure test specimens according to standard procedures provides valuable information for the engineer contemplating the selection of this material for the fabrication of a component meeting his performance requirement. This information, however, is not sufficient for the prediction of the component's structural performance in service, because standard procedures for testing of material specimens do not duplicate the stress field that will be generated in the specimen by the operational loading conditions.

To circumvent this shortcoming scale models of the component are fabricated and subjected to loads that will be encountered by the full size component in operation. If the model scale component performs satisfactorily the degree of confidence in the performance of the full size component will increase significantly. Since B₄C/Aluminum composite has previously never been applied to the construction of cylindrical external pressure housings it was deemed prudent to fabricate some model scale cylinders and evaluate their structural performance prior to committing the program to the fabrication of large cylindrical housings.

OBJECTIVE

The *primary objective* of the program for the fabrication and subsequent pressure testing of the model scale cylinders was to evaluate the structural performance of the B₄C/Aluminum composite in a complex stress field generated by operational pressure loading in monocoque cylinders.

The *secondary objective* of the program was to identify and solve any problems associated with the fabrication of monocoque cylinders from B₄C/Alumina composite.

The scope of the program was limited to two types of 6 inch diameter monocoque cylinders. Type 1 cylinders (figure B-1) were dimensioned for 215,000 psi nominal hoop stress and 50 percent buckling overload at 9,000 psi design pressure. Type 3 cylinders (figure B-2) were dimensioned for 132,000 psi nominal hoop stress and 100 percent buckling overload at 9,000 psi.

INSTRUMENTATION

All of the cylinders were straingaged with 0.25 inch rectangular straingage rosettes. A minimum of 5 rosettes was bonded to the interior surface at midbay to serve as a sensitive buckling detector. Some gages were also bonded to the interior surface near the ends of the cylinder to measure the magnitude of strains generated by the bulkheads (figures B-3 and B-4).

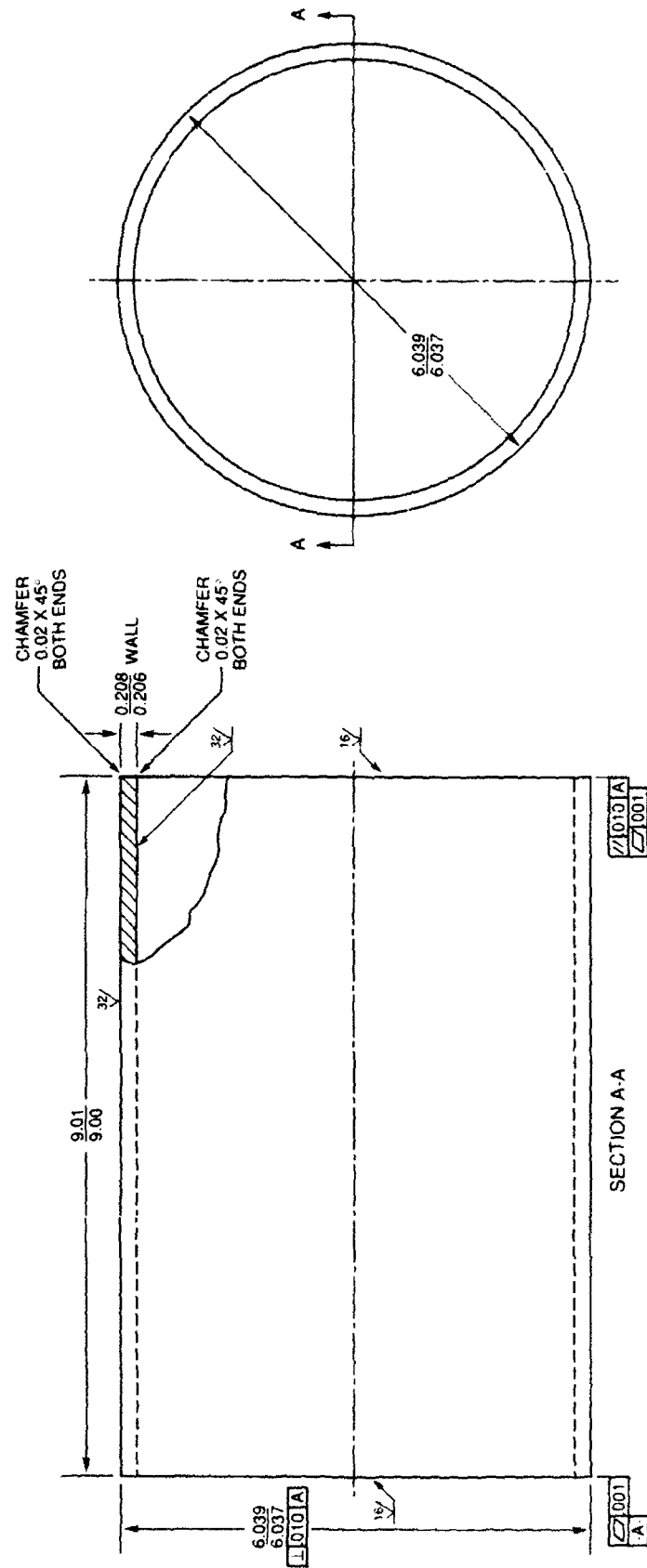


Figure B-1. Boron carbide aluminum cermet model scale--Type 1 housing.

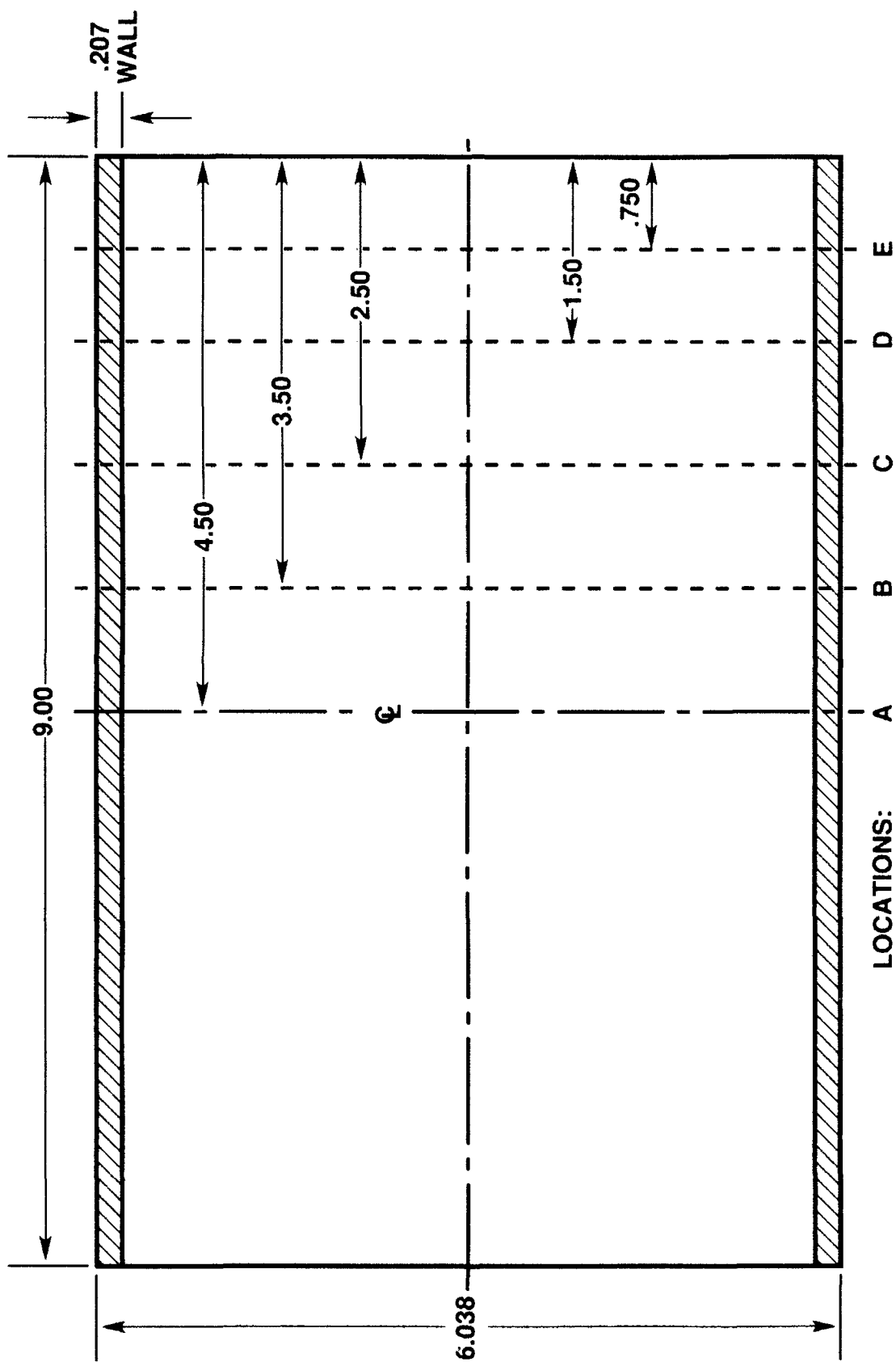


Figure B-3. Locations of gages on the interior of Dow Type 1 test cylinders—PN 55910-0126847.

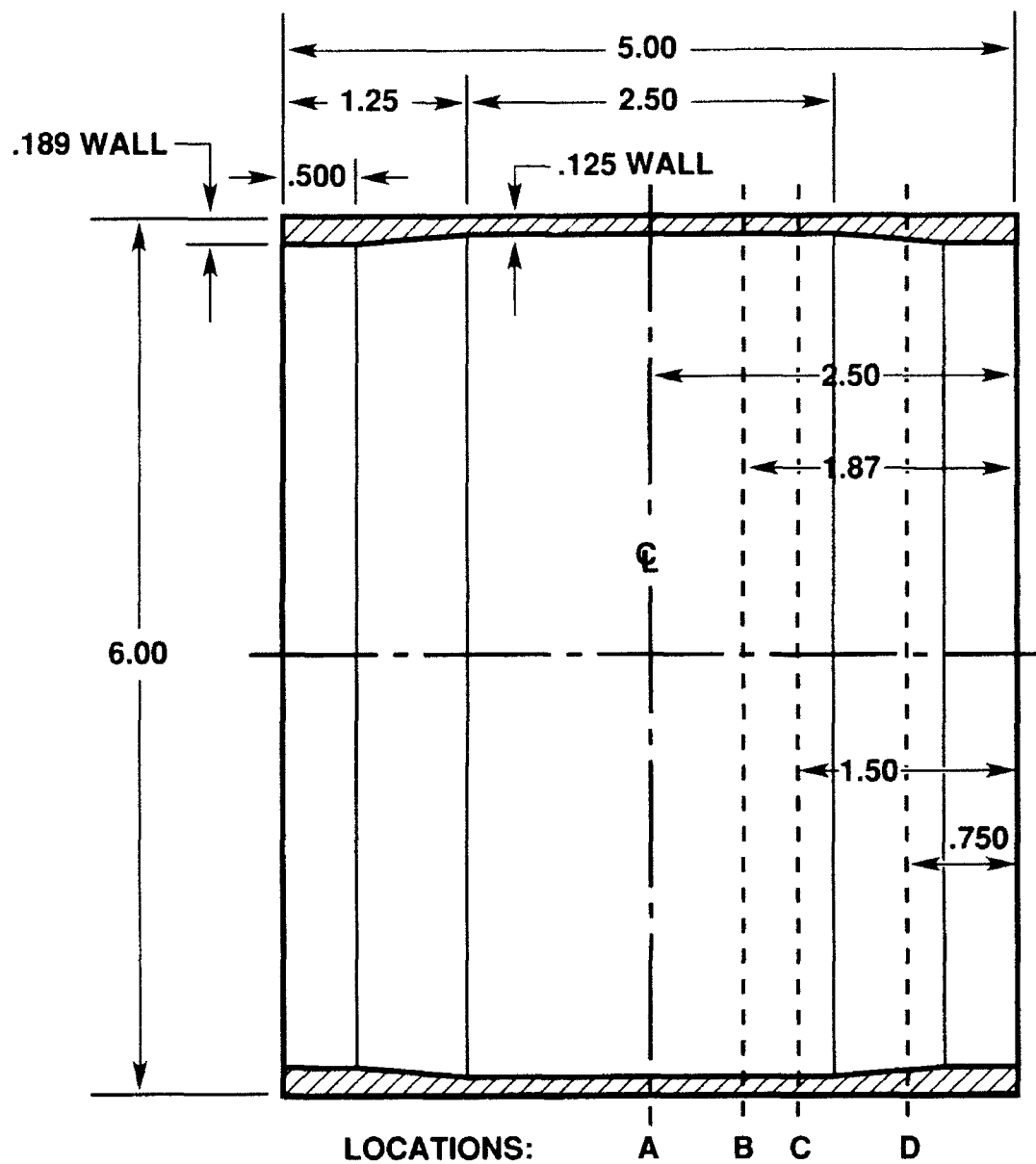


Figure B-4. Locations of gages on the interior of Dow Type 3 test cylinders—PN 55910-0126849.

Acoustic emission detectors were utilized to detect acoustic events during pressurization of the cylinders. In some cases they were bonded to the interior surface of the cylinders at midbay, while in other cases they were bonded to the exterior surface of the pressure vessel.

TEST SETUP

Prior to testing the ends of the cylinders were encased in epoxy filled titanium joint rings (figures B-5, B-6 and B-7) to protect them from chipping, and to reduce spalling on the plane bearing surfaces during repeated pressurization. The cylinders were pressure tested after being mated either with titanium hemispherical bulkheads, or steel plane bulkheads, both providing radial and axial support to the ends of the cylinders (figures B-8 and B-9).

The *steel plane bulkheads* were used in those tests where the primary objective of the test was to determine the maximum critical pressure attainable with a model cylinder of given dimensions (figure B-10). As a result the peak nominal hoop stresses and critical pressures generated by Type 1 and Type 3 cylinders supported by plane bulkheads were always significantly higher than when identical cylinders were supported at the ends by hemispherical bulkheads.

The *titanium hemispherical bulkheads* were employed only in those tests where the primary objective of the tests was to determine experimentally the effect of different fabrication parameters on the critical pressure of boron carbide aluminum composite monocoque cylinders mated to scale models of operational bulkheads (figures B-11 and B-12). Custom designed bulkhead penetrators were used for feeding through of instrumentation leads (figures B-13 and B-14).

TEST PROCEDURE

Pressure housings assembled from a single 6 inch diameter cylinder, and flat or hemispherical bulkheads (figures B-15 and B-16) were proof-tested twice to 10,000 psi, while the strain and acoustic emission readings were recorded at 1,000 psi intervals. Following these proof-tests the cylinders would be either pressure cycled to 9,000 psi, or pressurized to implosion.

In some tests (Test cylinders SN #5, 6 and 10) the pressurization was terminated prior to catastrophic implosion, when incipient buckling was detected by observing the departure of hoop strains from linearity at midbay. As a signal for termination of pressurization served the reversal of strain rate by any one of the strain gages oriented in hoop direction. Subsequently these test cylinders were mated with different bulkheads and were tested to catastrophic failure. In all cases the critical pressure was reached only when the test cylinders were equipped with plane steel bulkheads (figure B-17). Because of the high dynamic pressures generated by implosions the testing was conducted in surplus gun barrels converted into pressure vessels (figure B-18).

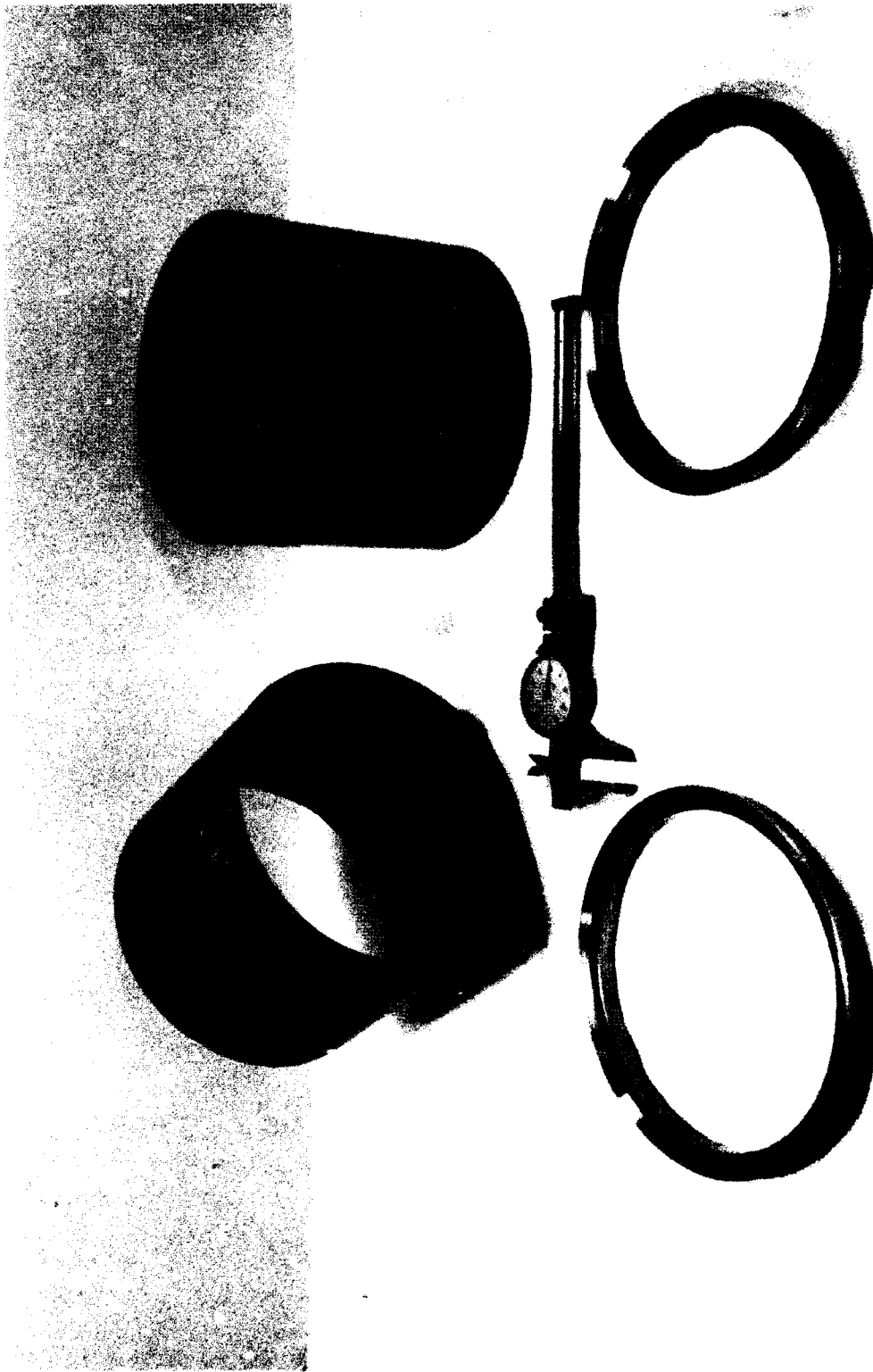
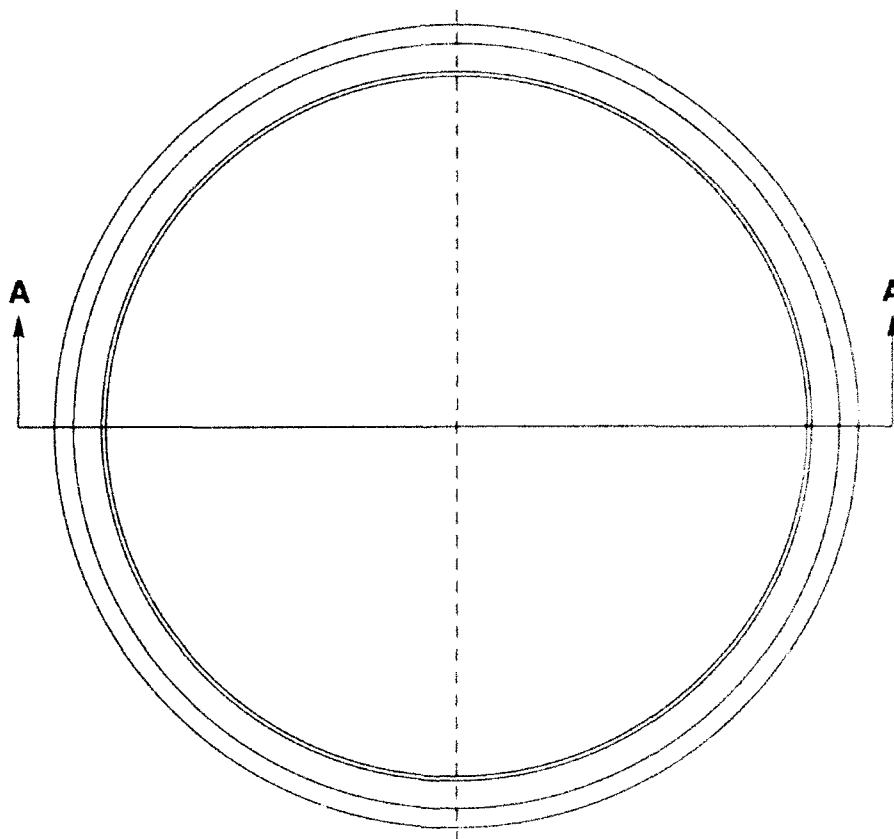
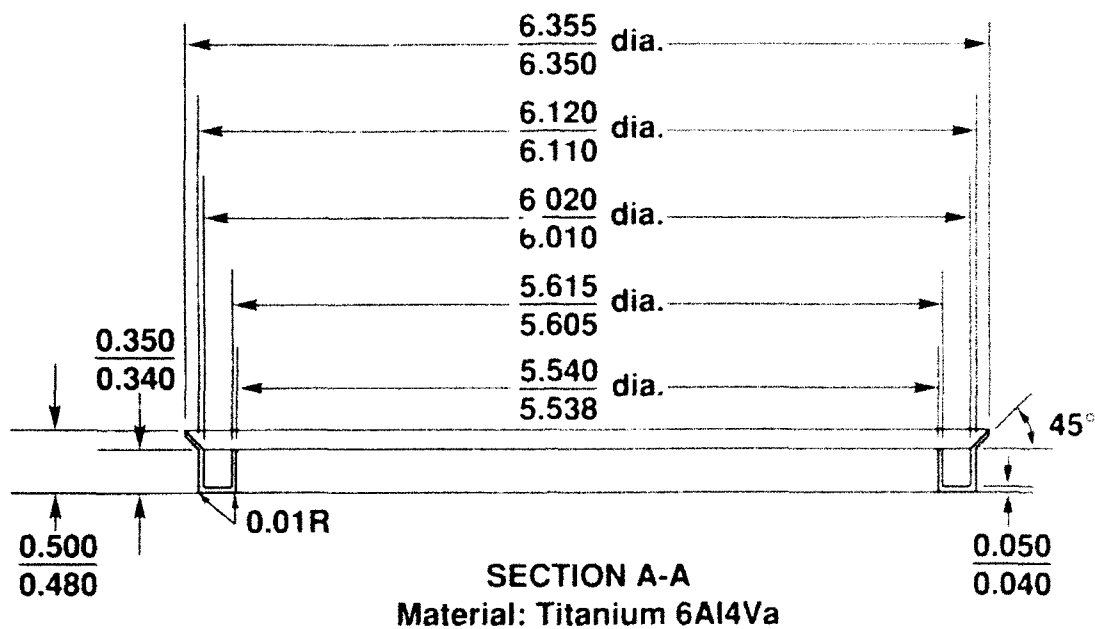
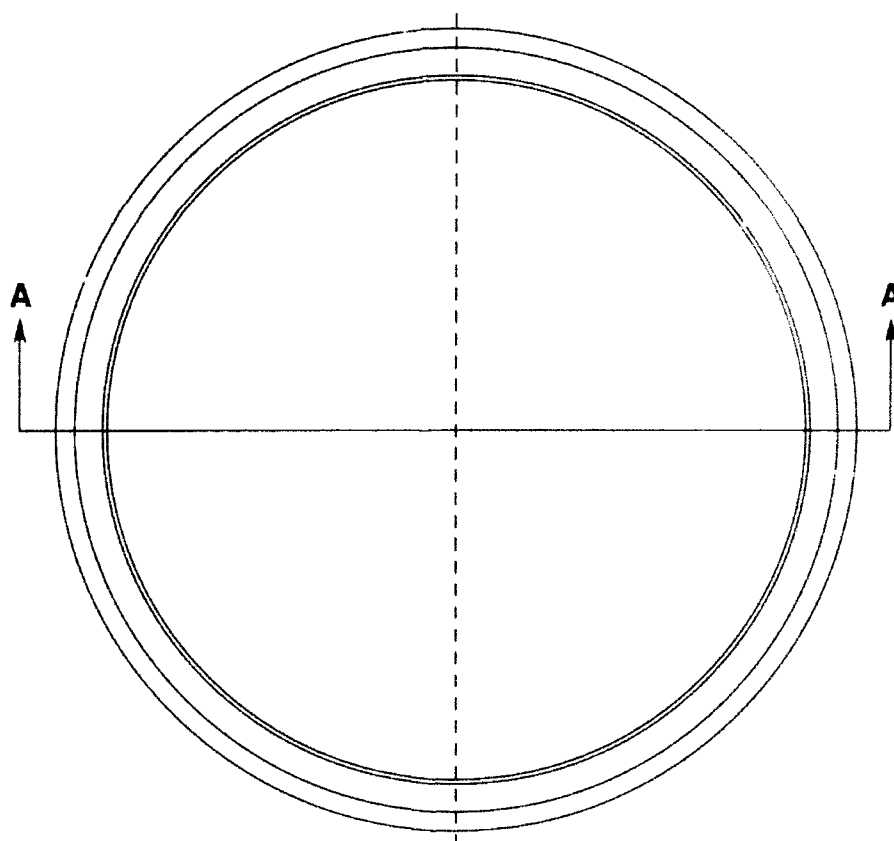
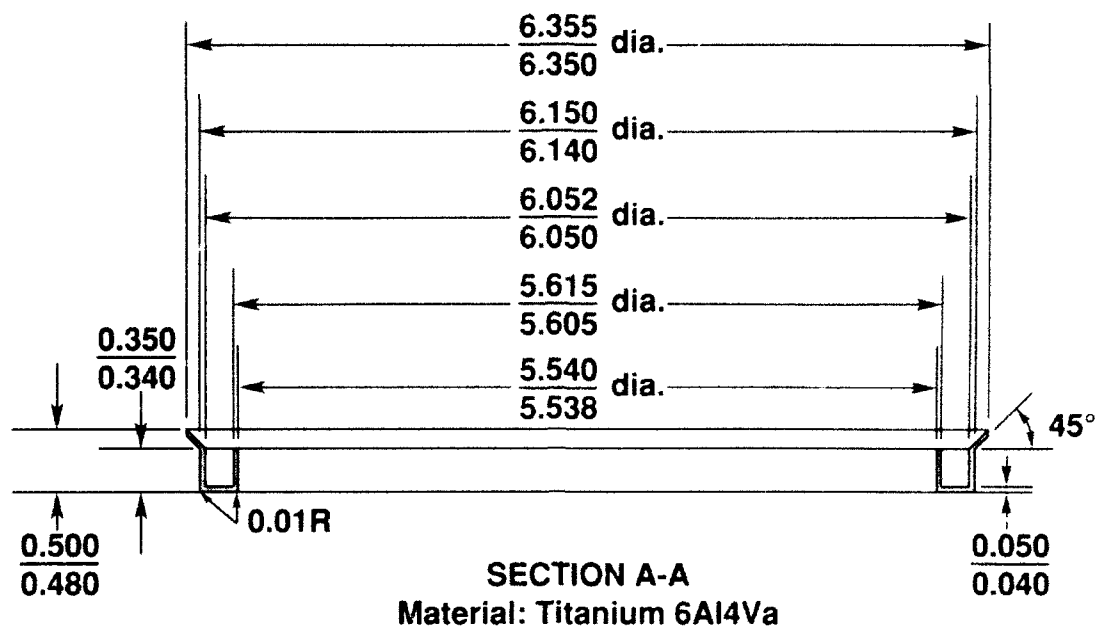


Figure B-5. Type 3 cylinders with titanium end caps prior to bonding.



ALL MEASUREMENTS IN inches

Figure B-6. End cap Mod 1 for model scale cylinders—6.0 inch outside diameter × 0.19 inch thickness.



ALL MEASUREMENTS IN inches

Figure B-7. End cap Mod 1 for model scale cylinders—6.04 inch outside diameter \times 0.2076 inch thickness.

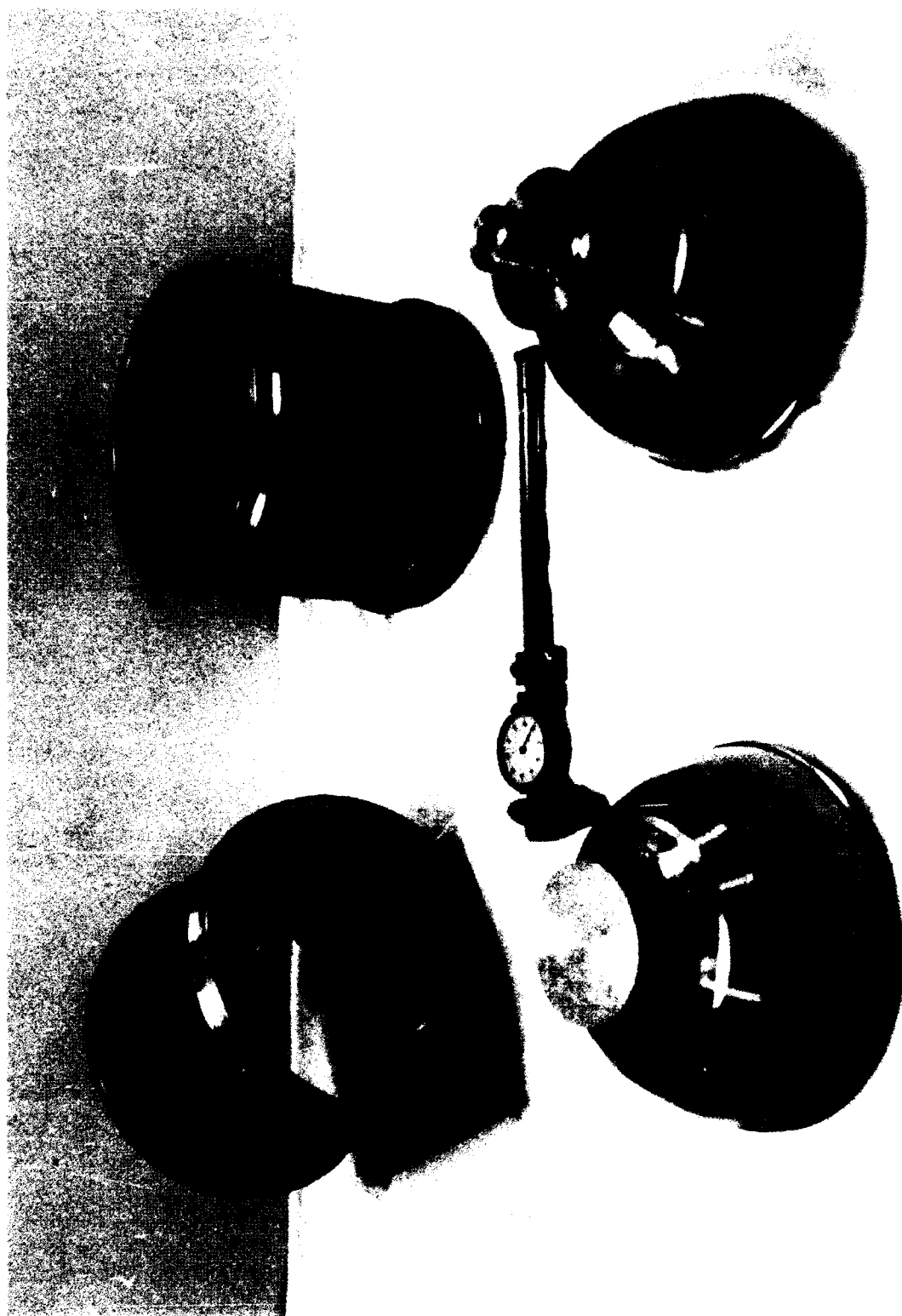


Figure B-8. Type 3 cylinders with bonded on end caps prior to mating with hemispherical bulkheads for cyclic pressure testing.

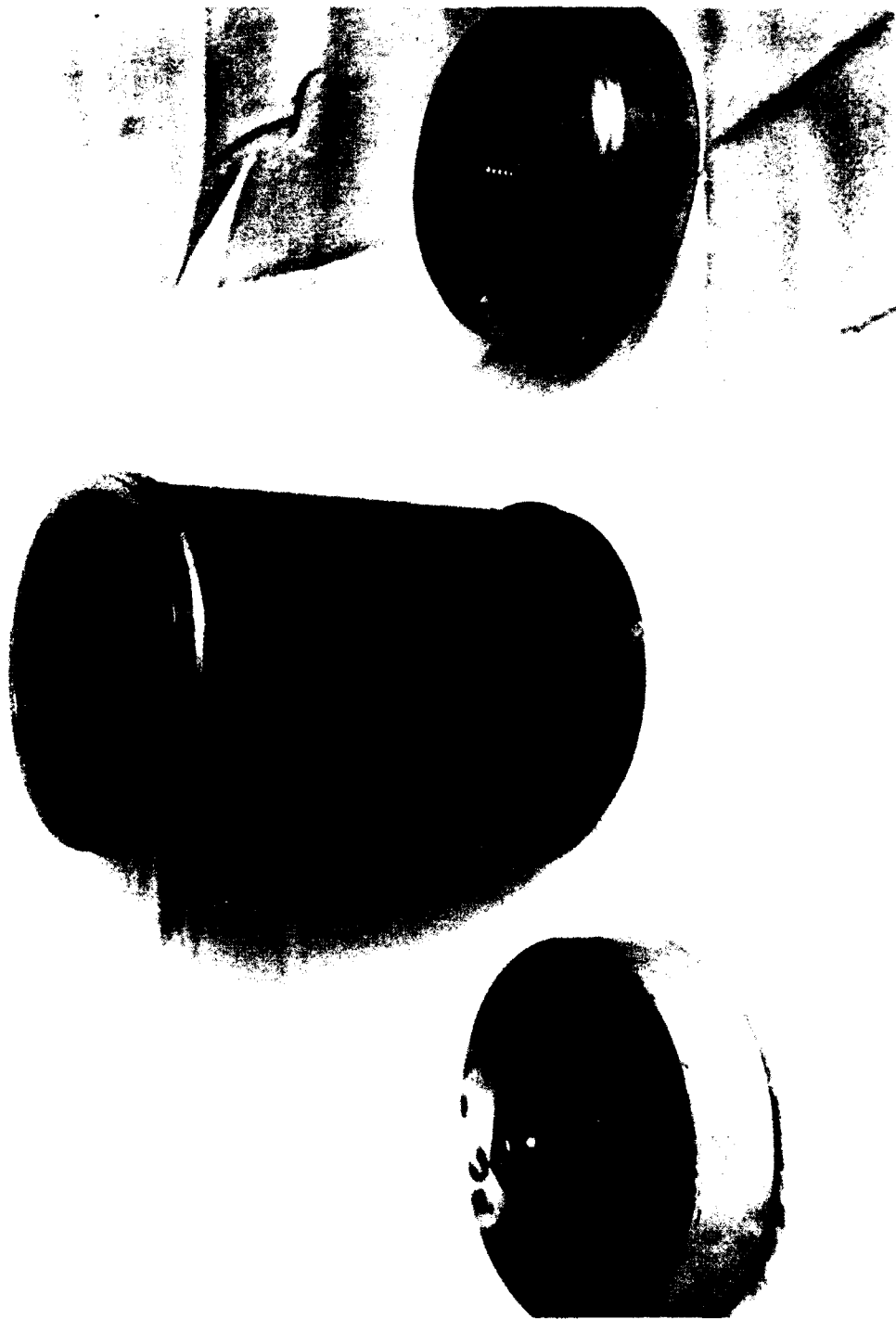


Figure B-9. Type 1 cylinder with bonded on end caps prior to mating with plane steel bulkheads for implosion testing.

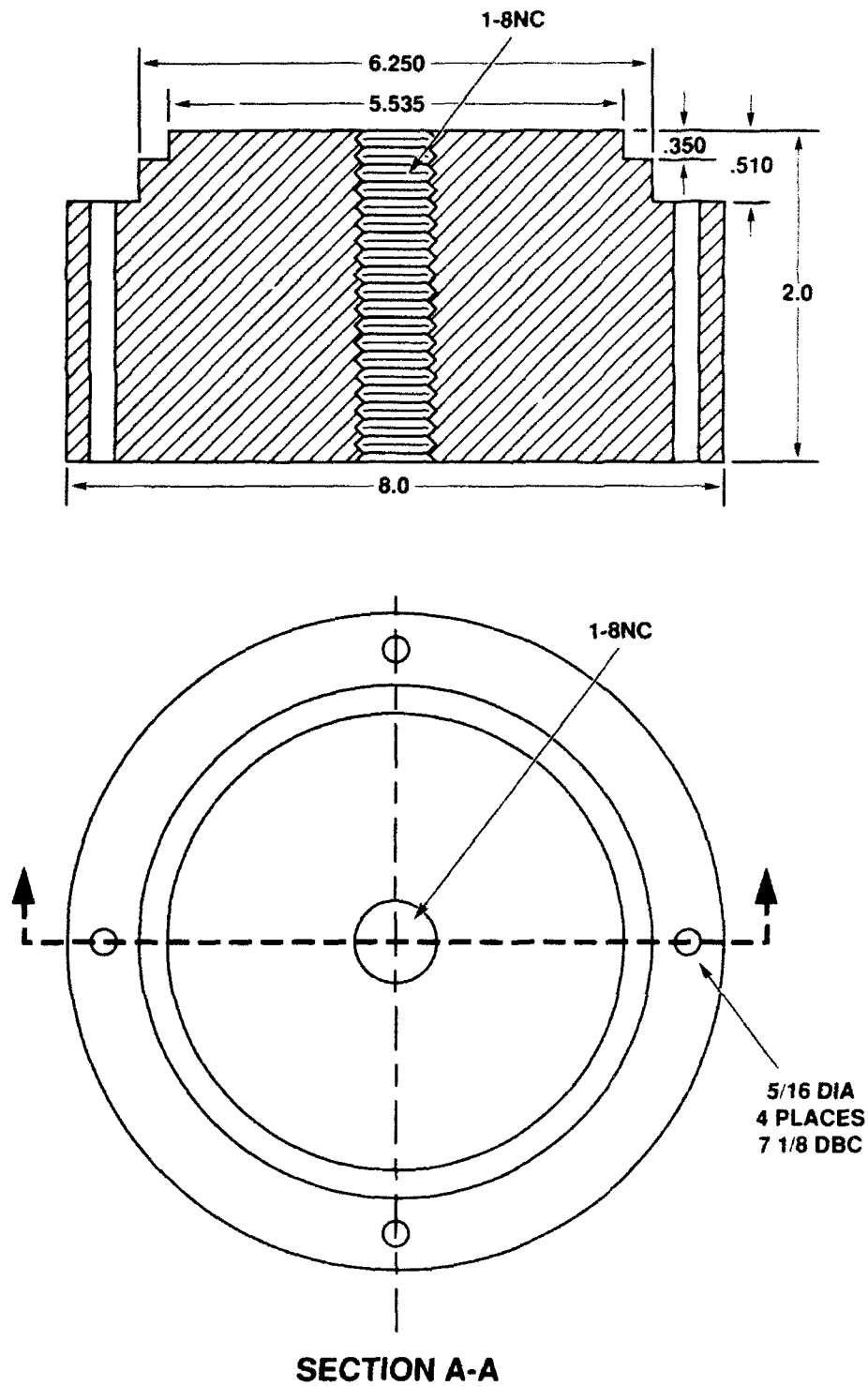


Figure B-10. Plane steel bulkhead for Type 1 and 3 cylinders.

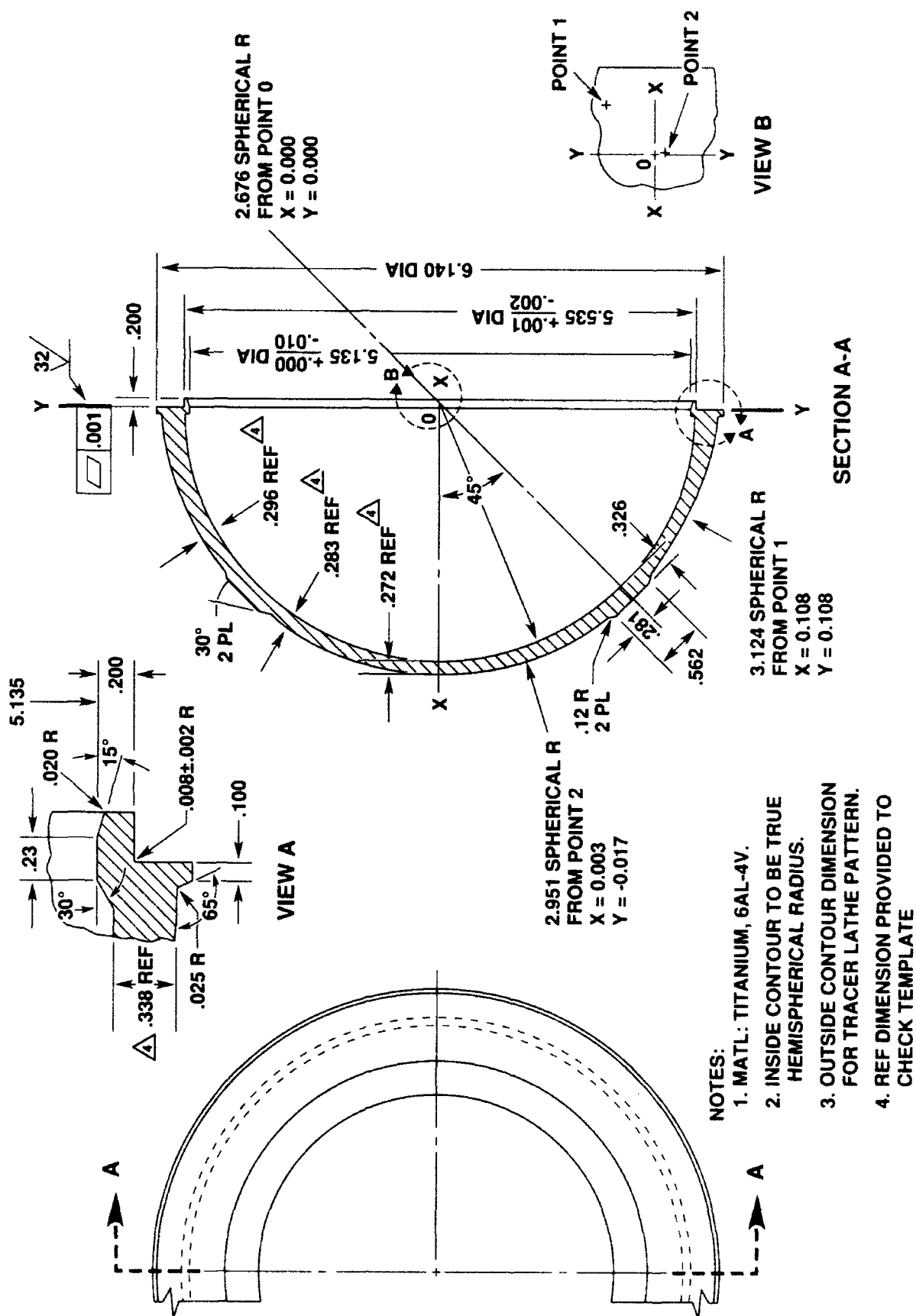


Figure B-11. Titanium end closure Model 2.

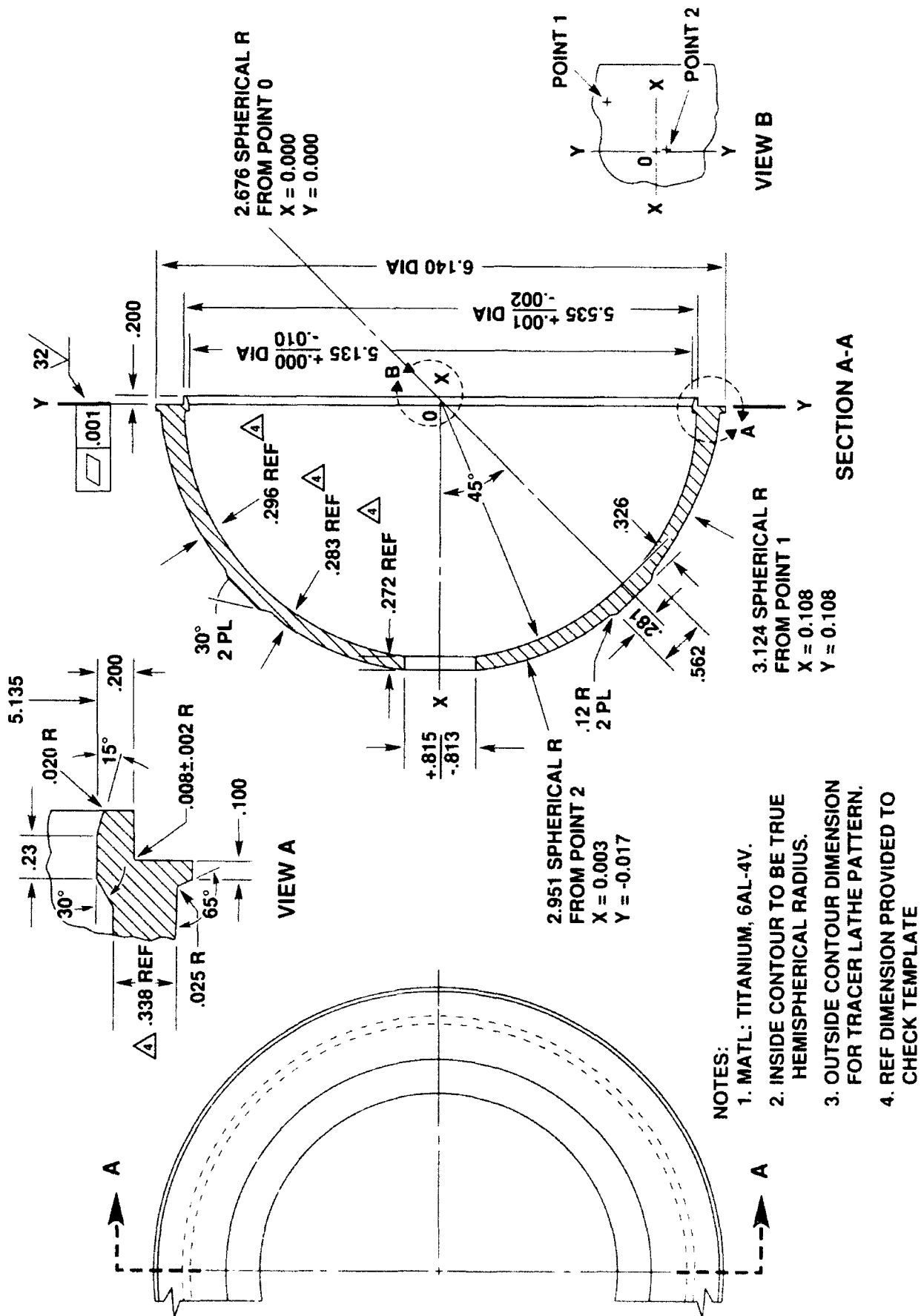


Figure B-12. Titanium end closure Model 2 with penetrations.

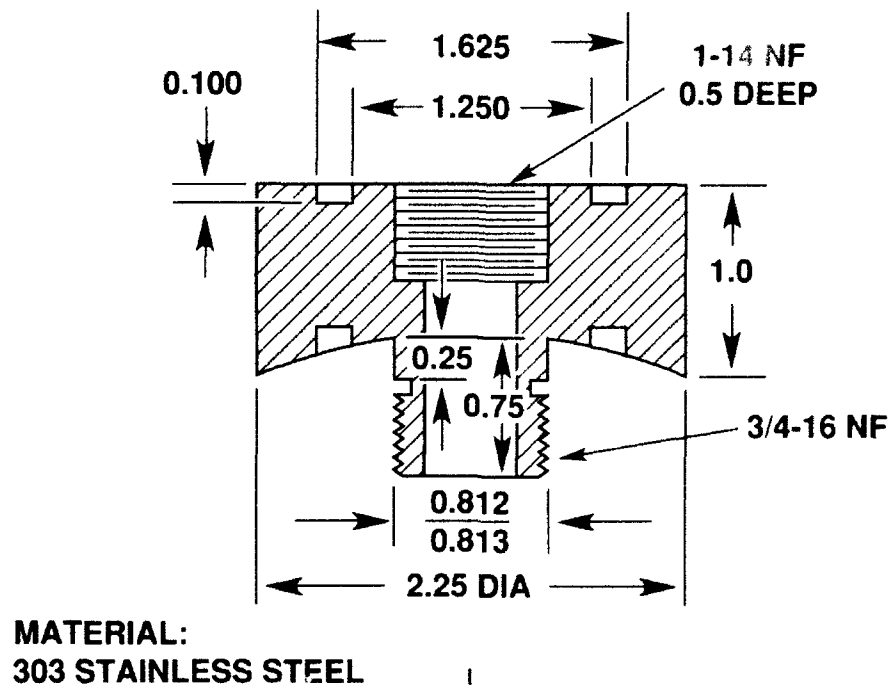
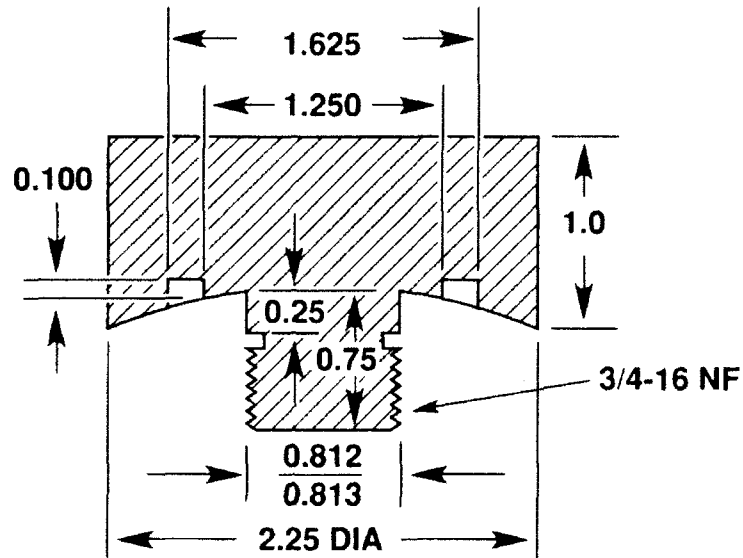


Figure B-13. Spherical bulkhead penetrator.



MATERIAL:
303 STAINLESS STEEL

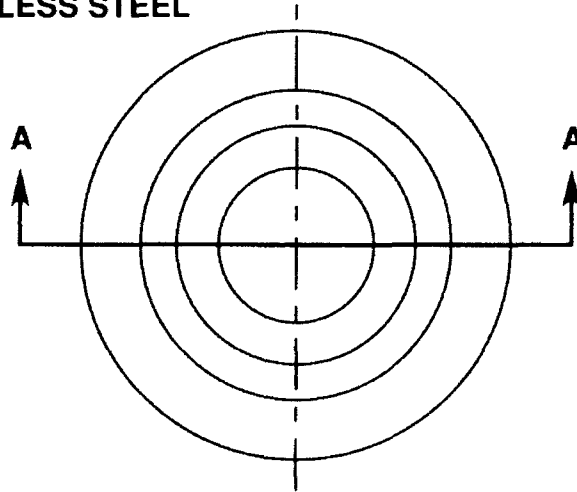


Figure B-14. Spherical bulkhead penetration plug.



Figure B-15. Instrumented Type 3 cylinder equipped with hemispherical bulkheads.

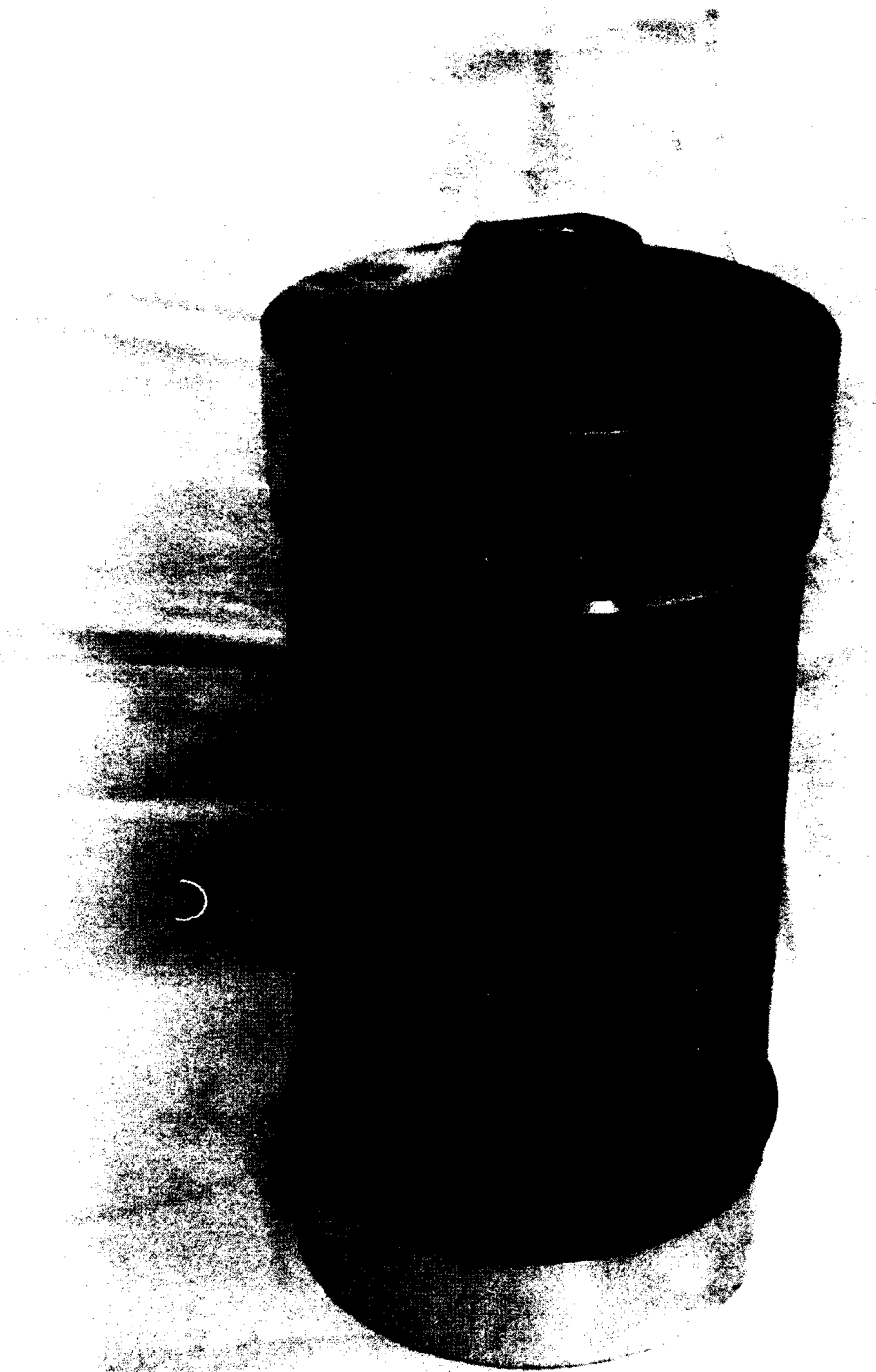


Figure B-16. Type 1 cylinder equipped with plane steel bulkheads. Critical pressure of cylinders tested with plane bulkheads was always higher than cylinders tested with hemispherical bulkheads.

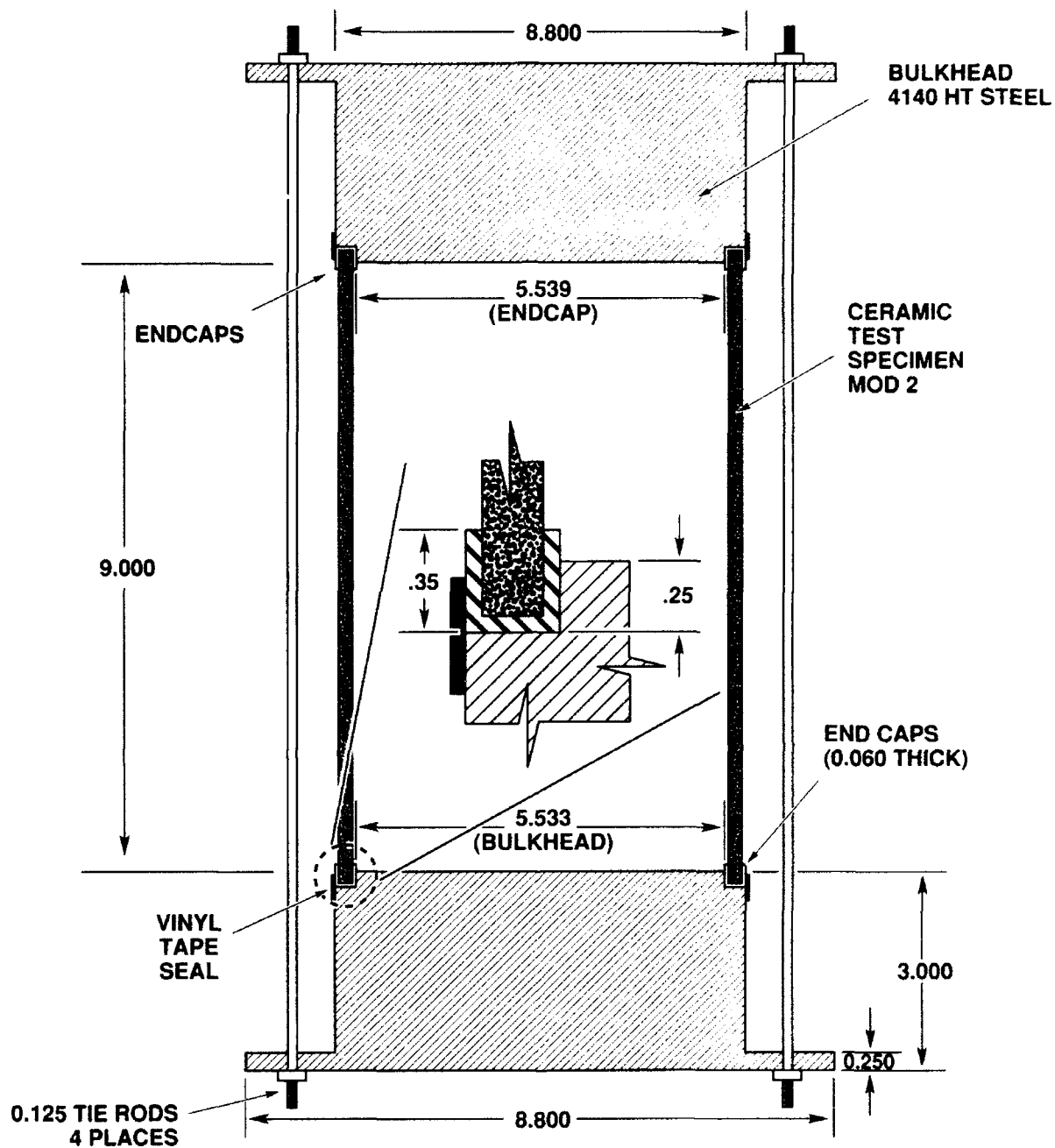


Figure B-17. Test arrangement for implosion testing of 6-inch ceramic cylinders.

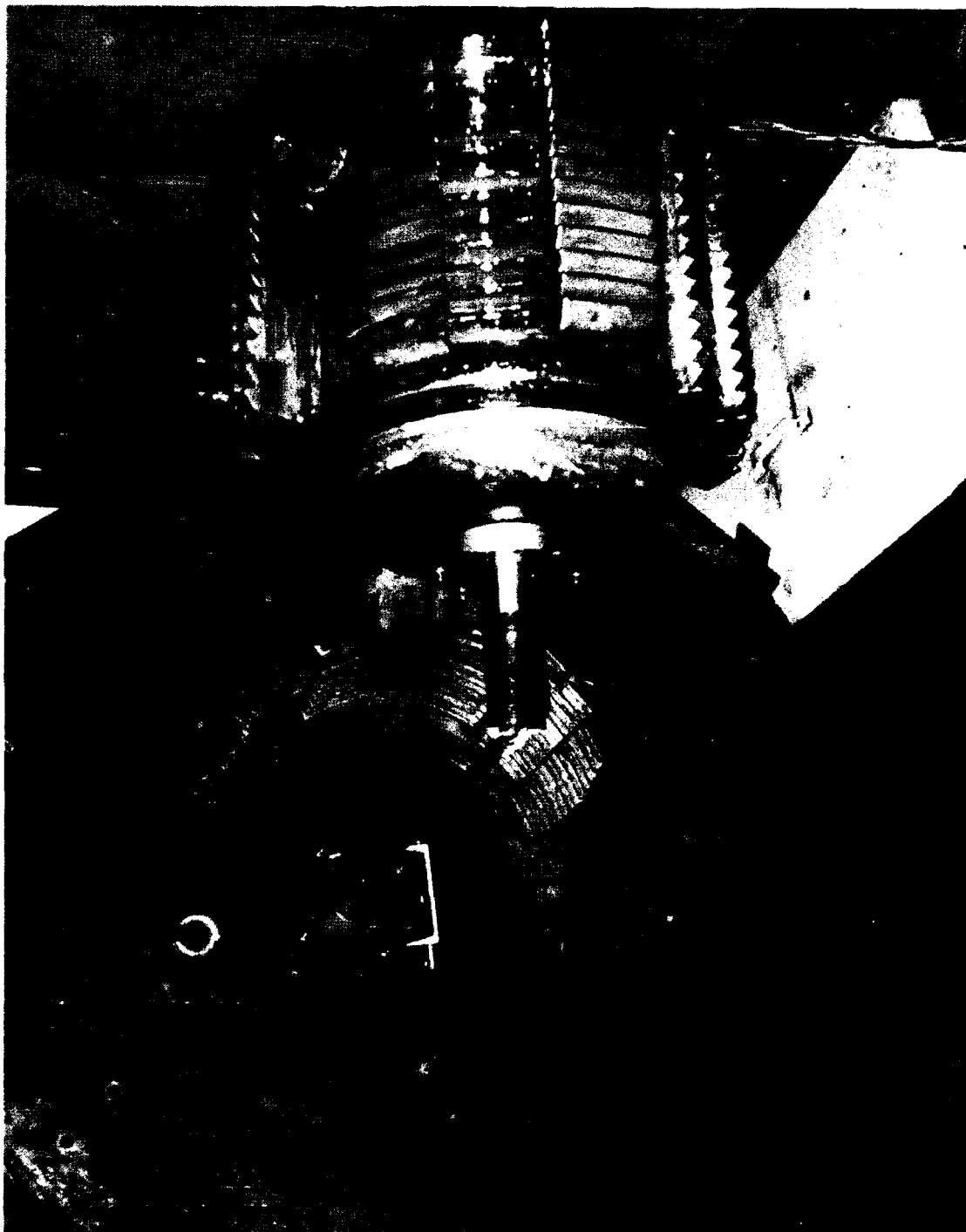


Figure B-18. Pressure vessel used for implosion testing of ceramic cylinders.

TEST RESULTS

1. Cylinders radially supported at the ends by plane bulkheads failed at higher pressures than identical cylinders radially supported at the ends by hemispherical bulkheads. The increase in critical pressure was in the range of 15 to 20 percent.
2. All of the cylinders tested failed by mechanism of elastic instability (i.e., buckling), rather than by failure of material in compression. The modulus of elasticity of the ceramic composite with 70 percent B_4C in the 6-inch diameter cylinder bodies has been calculated on the basis of measured strains to fall into the 43 to 45×10^6 psi range. Although the compressive strength of this ceramic composite in the cylinder bodies has not been experimentally determined, it has been shown to exceed 300,000 psi.
3. The cyclic fatigue life of the bearing surfaces on the 6-inch diameter ceramic composite cylinders has been determined to exceed 3,000 pressure cycles at axial bearing pressure of 66,000 psi. During these tests the ends of the cylinders were resting on a 0.010 inch thick layer of epoxy held captive between mating surfaces of the ceramic composite cylinder and the titanium joint ring.
4. Type I monocoque cylinder with $L/D_o = 1.5$, when fabricated from B_4C aluminum composite, equipped with titanium joint rings, and supported by rigid bulkheads can serve as a housing with a 0.36 weight to displacement ratio for 9,000 psi (20,000 feet) operational pressure, while providing a safety factor of 2.2 against implosion by elastic instability, or material failure.
5. When Type I monocoque cylinder is mated with the lightweight Model I titanium hemispheres (figure B-19) developed in a previous NRaD program the resultant weight to displacement ratio of the housing as well as its resultant safety factor are calculated to be 0.46 and 1.25 respectively.

With Model 2 titanium hemispheres (figures B-11 and B-12) the resultant weight to displacement ratio, as well as its resultant safety factor of the housing are calculated to be 0.65 and 1.73, respectively.

With Model 3 ceramic composite hemispheres (figure B-20) equipped with titanium mounting ring (figure B-21) the resultant weight to displacement ratio, as well as the resultant safety factor of the housing are calculated to be 0.35 and 2, respectively.

CONCLUSIONS

1. Monocoque cylinders with $t/D_o = 0.034$ and $L/D_o = 1.5$ dimensions when equipped with titanium joint rings provide a weight to displacement factor of 0.36 at a safety factor of 2.2 for operational depth of 20,000 feet when fabricated from DOW ceramic aluminum composite containing 70 percent boron carbide (Figure 22A).
2. When the above cylinders are mated with the aid of joint rings to hemispheres of the same ceramic composite the weight to displacement ratio of the whole housing decreases to 0.35 without any reduction in the magnitude of safety factor.

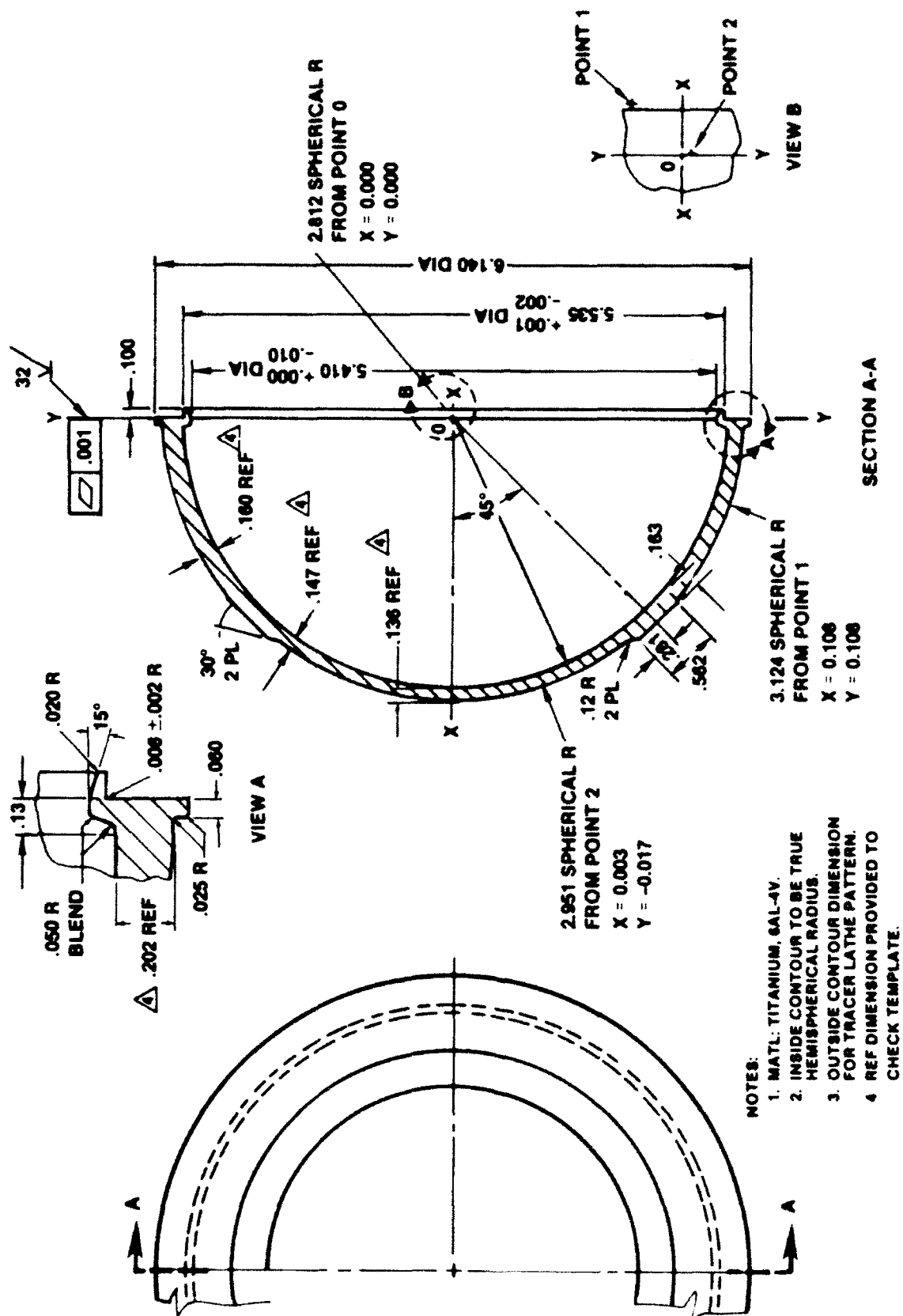


Figure B-19. Titanium end closure, Model 1.

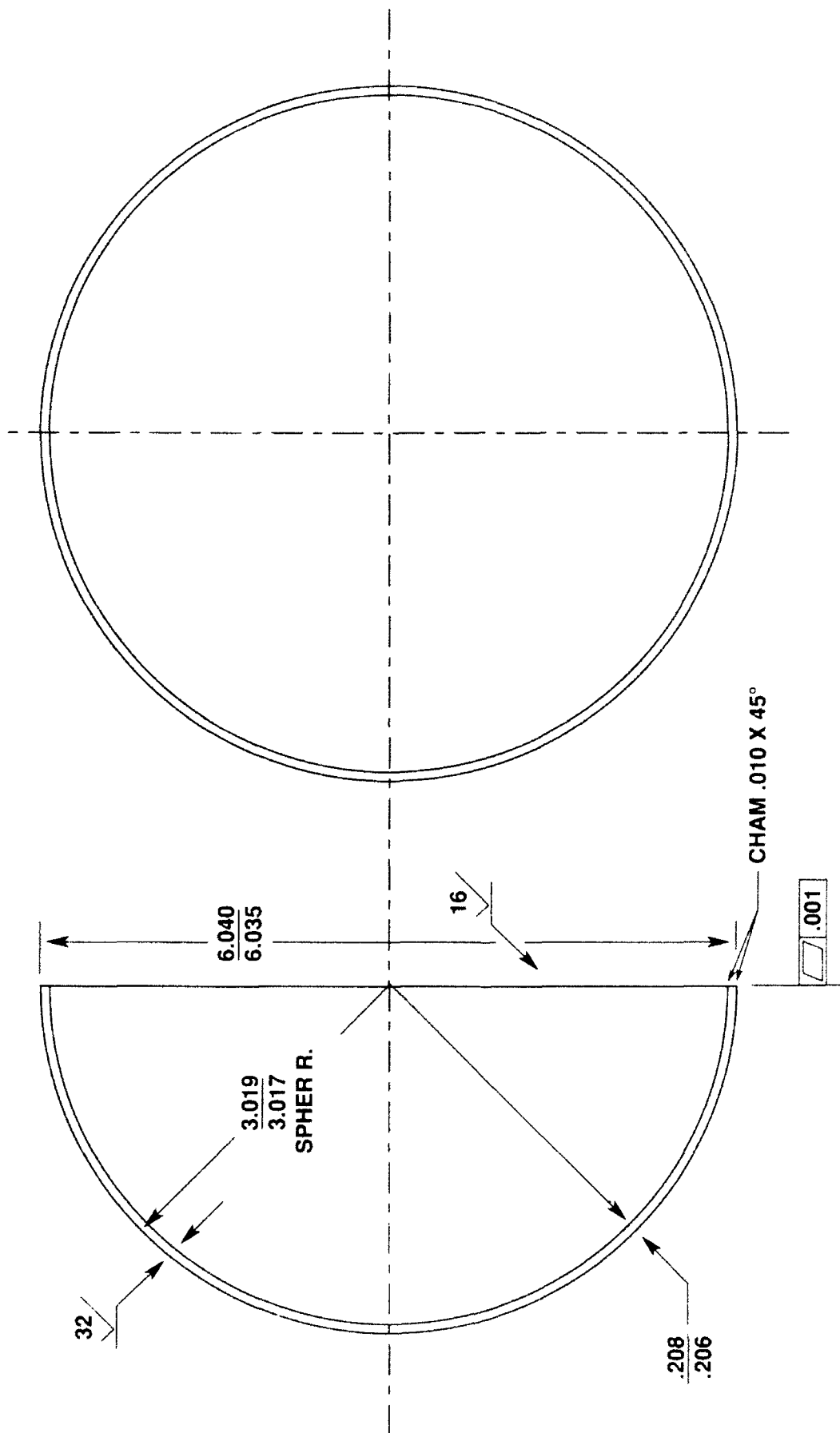
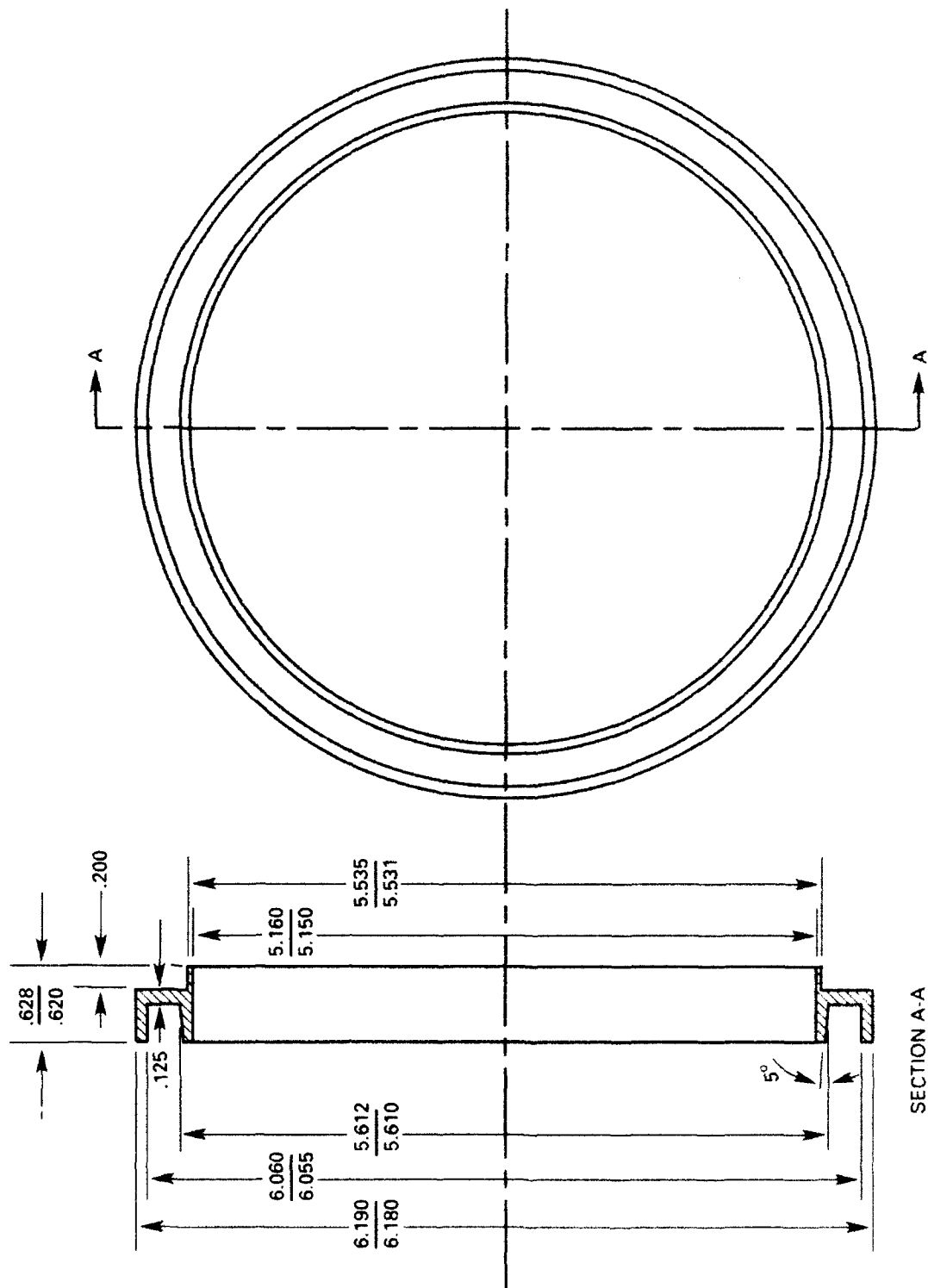


Figure B-20. Ceramic end closure, Model 3.



MATERIAL: TITANIUM ALLOY 6Al4Vα

Figure B-21. Ceramic hemisphere mounting ring.

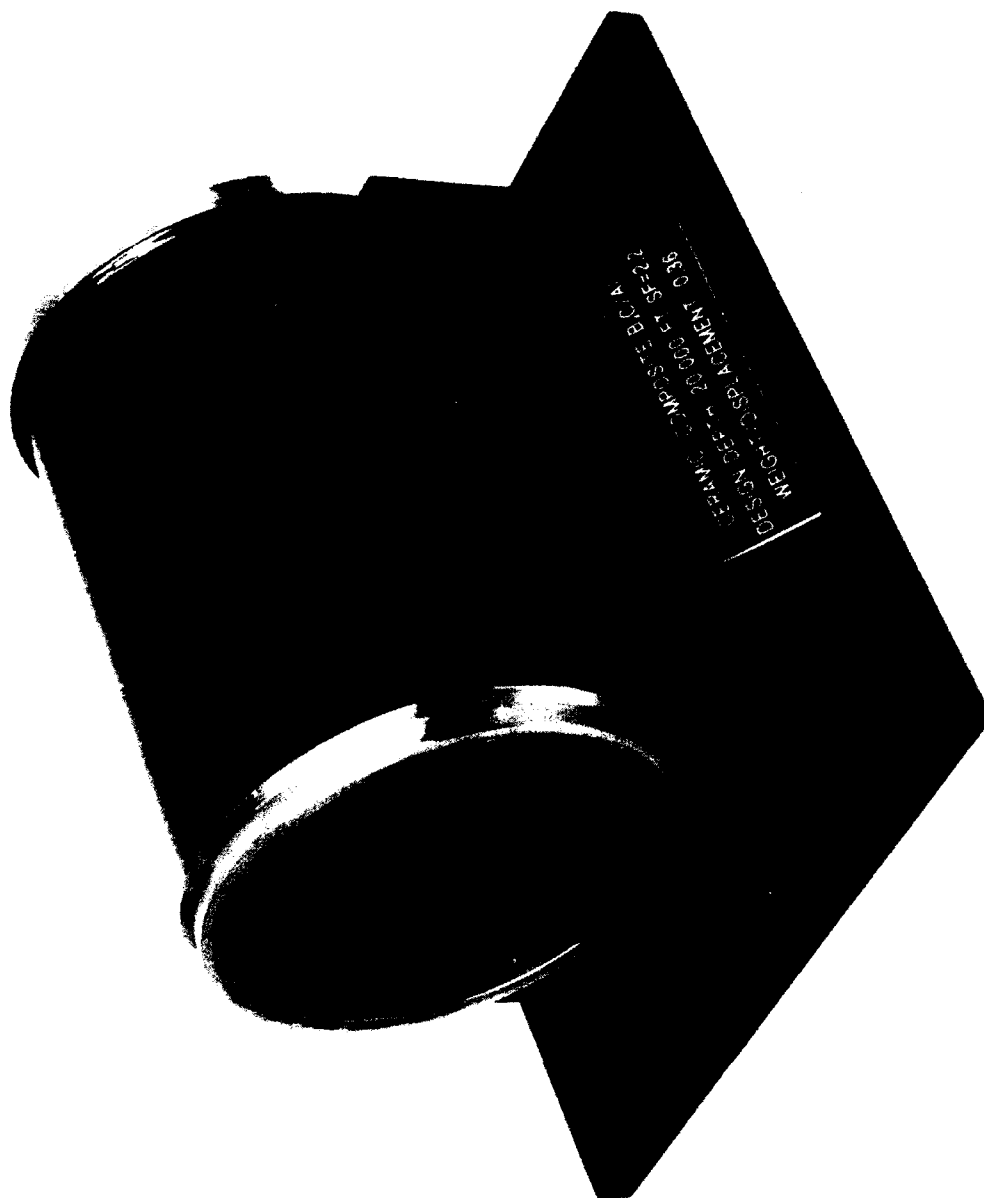


Figure B-22. Type 1 ceramic composite cylinder successfully withstood 19,500 psi external pressure when tested with plane steel bulkheads. When tested with spherical titanium bulkheads it imploded at 15,700 psi.

Test Cylinder SN#1 Type 3

Table 1 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 1
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location A - Midbay		Location B	
	1	2	3	4
	Hoop	Axial	Hoop	Axial
0	0	0	0	0
1000	-490	-137	-482	-166
2000	-999	-281	-988	-316
3000	-1554	-431	-1540	-468
4000	-2139	-591	-2120	-628
5000	-2744	-748	-2712	-780
6000	-3391	-911	-3358	-938
7000	-3995	-1063	-3962	-1082
8000	-4617	-1218	-4572	-1228
9000	-5226	-1372	-5162	-1361
10000	-6039	-1532	-5696	-1473

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick

Cylinder Weight: 597 grams

Catastrophic implosion 10,500 psi

Maximum compressive hoop stress at failure: 252,000 psi

Interior Gage Locations

NOTES: ALL strains are in microinches per inch

Electric resistance strain gages are CEA-06-125UT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick

Cylinder Weight: 597 grams

Catastrophic implosion 10,500 psi

Maximum compressive hoop stress at failure: 252,000 psi

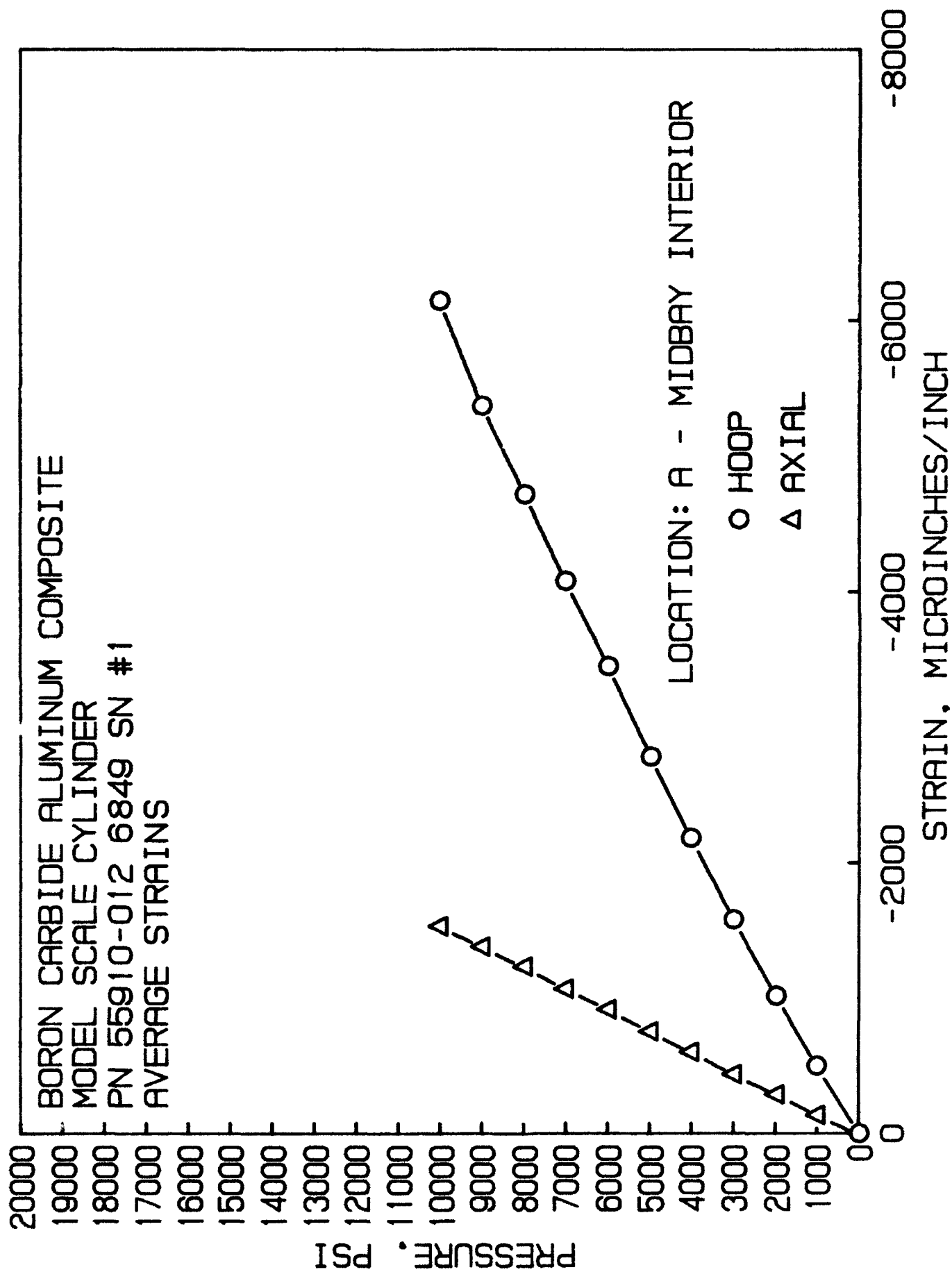
Test Date: 11-9-91

ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-012 6849 SN# 1

Pressurization # 1

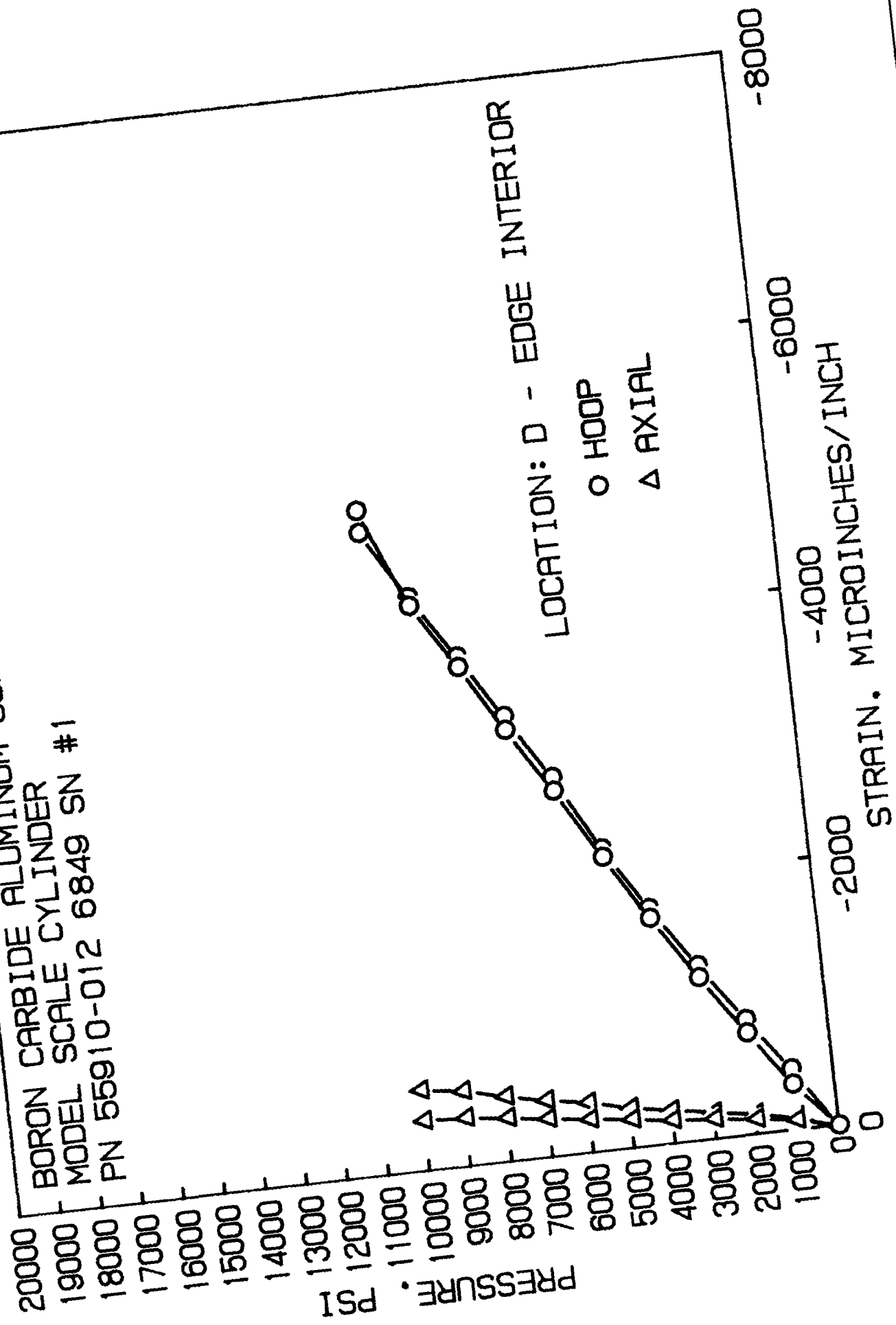
Pressure	Events	Time	Notes:
0000	0	7:35	1. Transducer: AET AC175
1000	1184-1195	7:40	SN# 7799 5 to 200 KHZ
2000	1268-1292	7:43	2. Amplifier Setting:
3000	1320-1331	7:45	Rate: T
4000	1381-1402	7:48	Gain: 80 DB
5000	1438-1466	7:51	Threshold: Automatic
6000	1579-1655	7:53	Function: Events
7000	1865-2181	7:55	3: Recorder:
8000	2732-3274	7:58	Channel "A" Events,
9000	3426-3746	8:01	4000 Full Range
10000	4870-6055	8:04	Channel "B" Rms,
10500	6327	8:05	50 MV Full Scale, 0.5 CM/Min Chart Scale

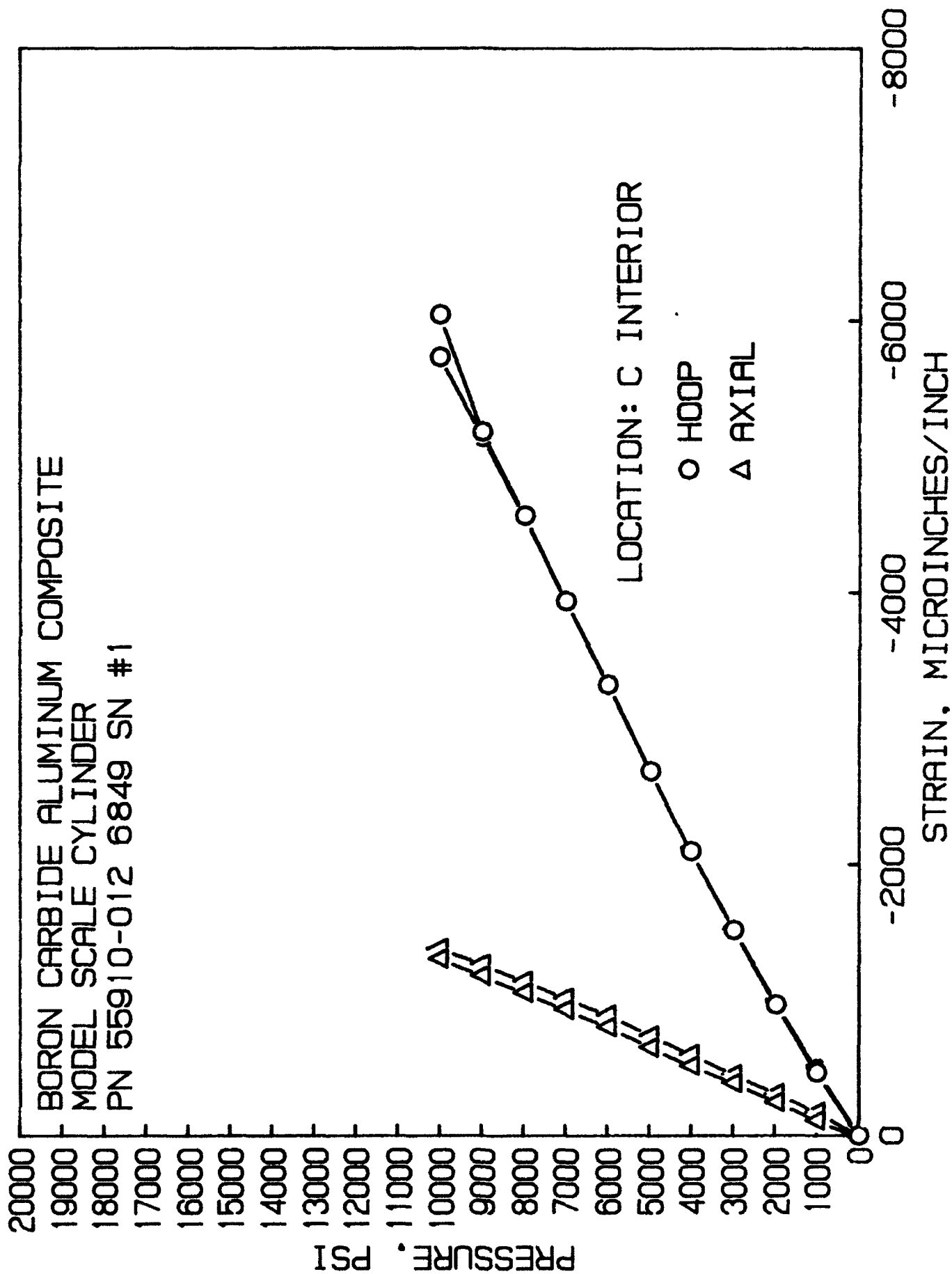
Failed at 10,500 psig

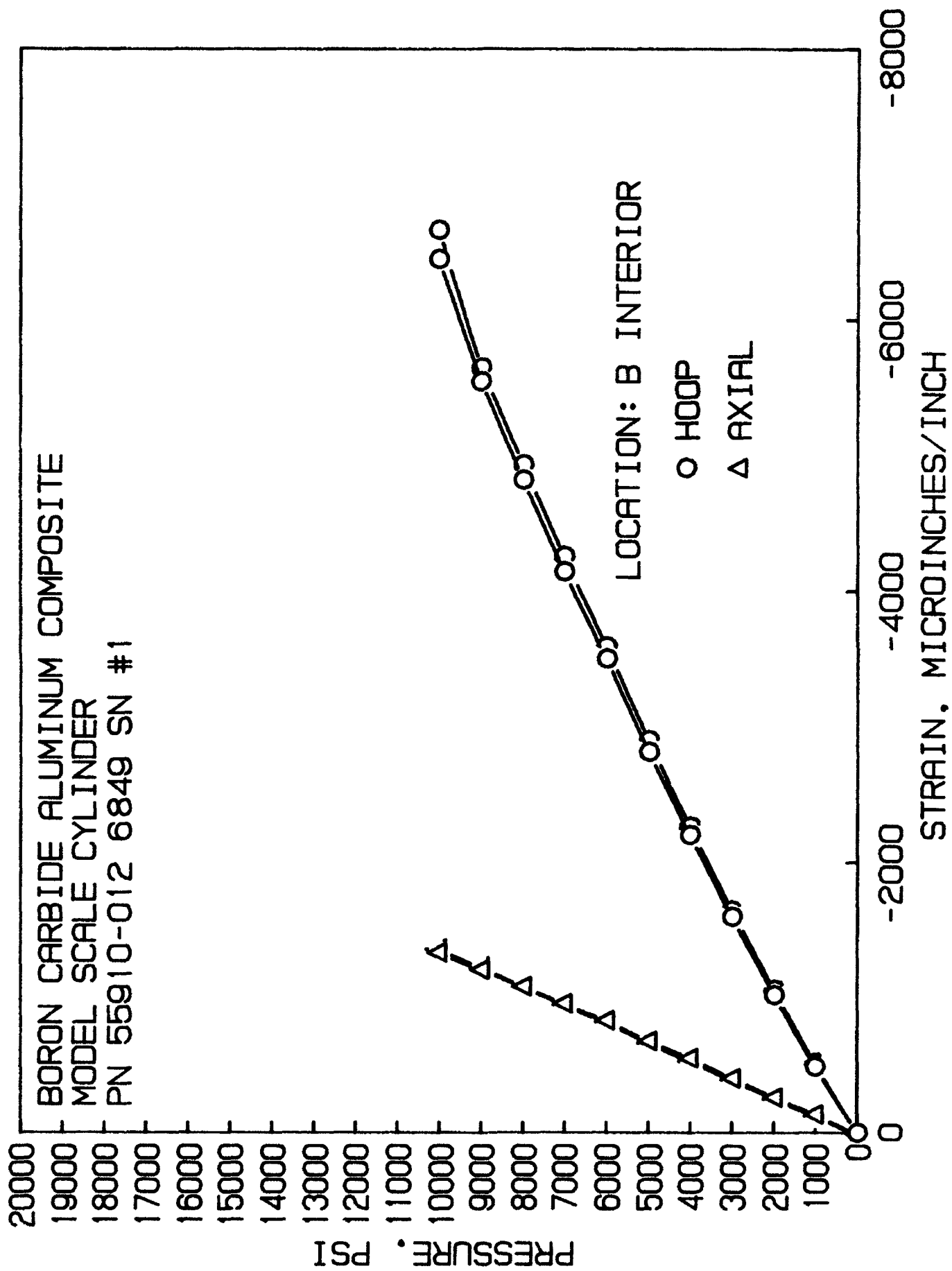


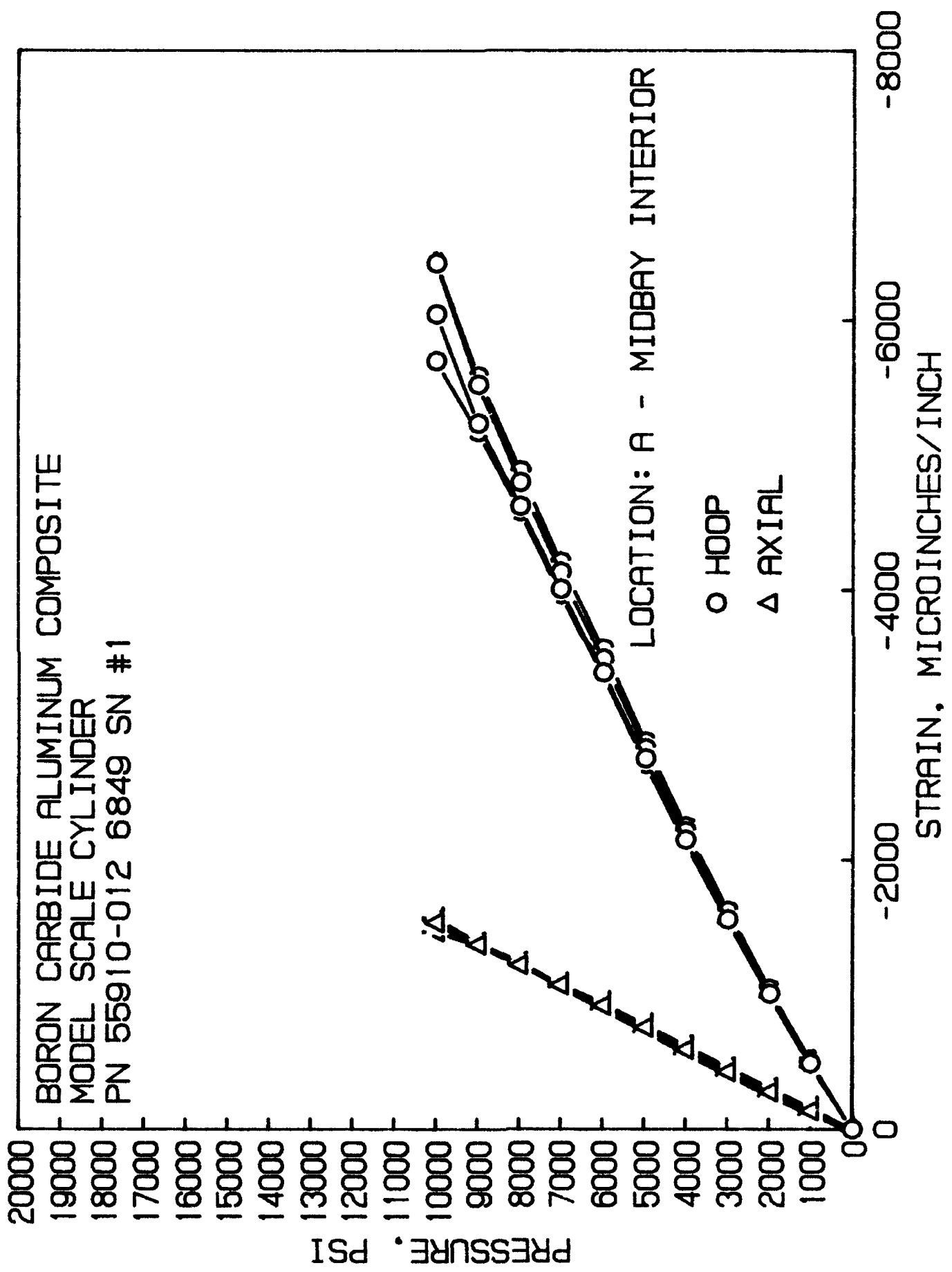
BORON CARBIDE ALUMINUM COMPOSITE

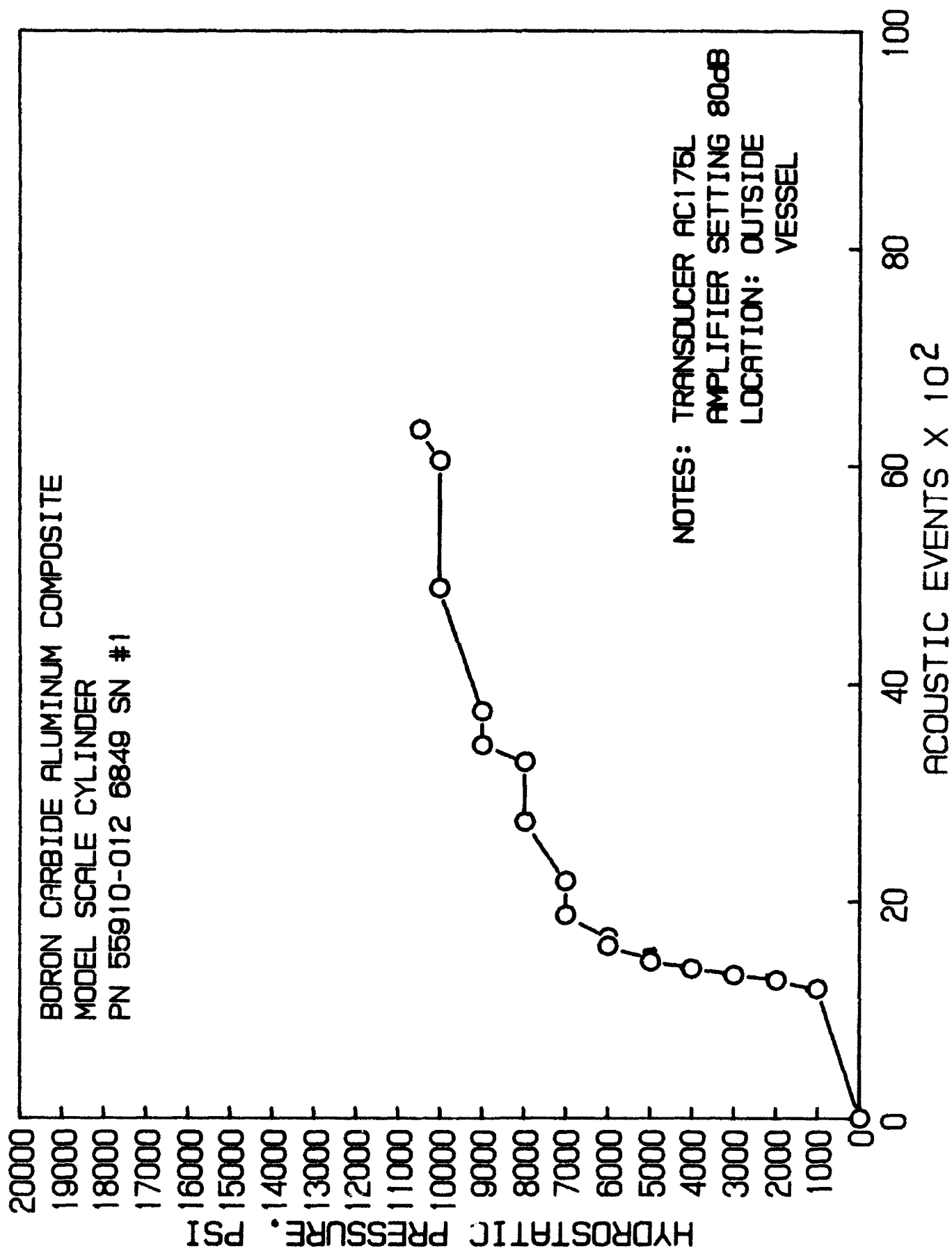
BORON CARBIDE ALUMINUM COMPOSITE
 MODEL SCALE CYLINDER #1
 PN 55910-012 6849 SN #1











Test Cylinder SN#2 Type 3

Table 2 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 2
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location A - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-432	-175	-481	-159	-446	-128	-450	-193	-495	-112
2000	-910	-332	-941	-312	-910	-278	-933	-343	-960	-261
3000	-1429	-501	-1439	-484	-1413	-442	-1459	-502	-1467	-424
4000	-1894	-652	-1890	-629	-1863	-586	-1932	-643	-1918	-568
5000	-2384	-810	-2363	-783	-2330	-738	-2427	-792	-2388	-721
6000	-2910	-980	-2869	-947	-2832	-901	-2959	-951	-2890	-886
7000	-3412	-1140	-3350	-1100	-3305	-1156	-3467	-1100	-3364	-1043
8000	-3923	-1302	-3839	-1255	-3781	-1214	-3980	-1248	-3842	-1202
9000	-4430	-1459	-4320	-1405	-4245	-1369	-4489	-1392	-4310	-1360
10000	-4958	-1616	-4779	-1555	-4638	-1533	-5019	-1526	-4795	-1523
11000	-5517	-1769	-5234	-1710	-5034	-1710	-5579	-1660	-5314	-1691
12000	-6225	-1902	-5580	-1878	-5212	-1917	-6197	-1781	-6094	-1830
12600	-7631									

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125UT-350, Gage Factor 2.09
Ceramic Composition: Boron Carbide Aluminum Composite
End Closures: Titanium Hemispherical Bulkheads providing radial support
Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick
Cylinder Weight: 597 grams
Catastrophic Implosion 12,600 psi
Maximum compressive hoop stress at failure: 302,400 psi

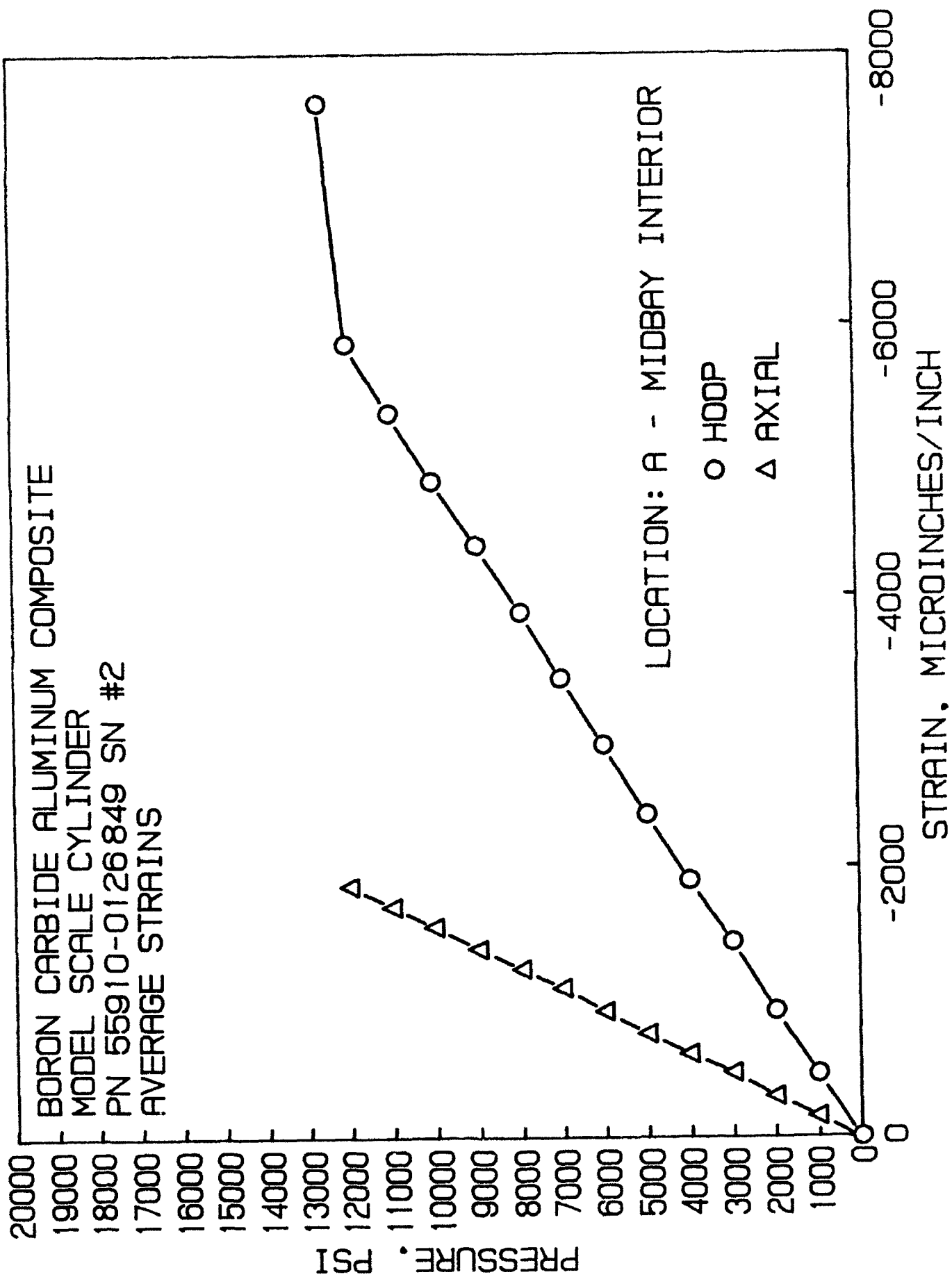
Test Date: 12-14-91

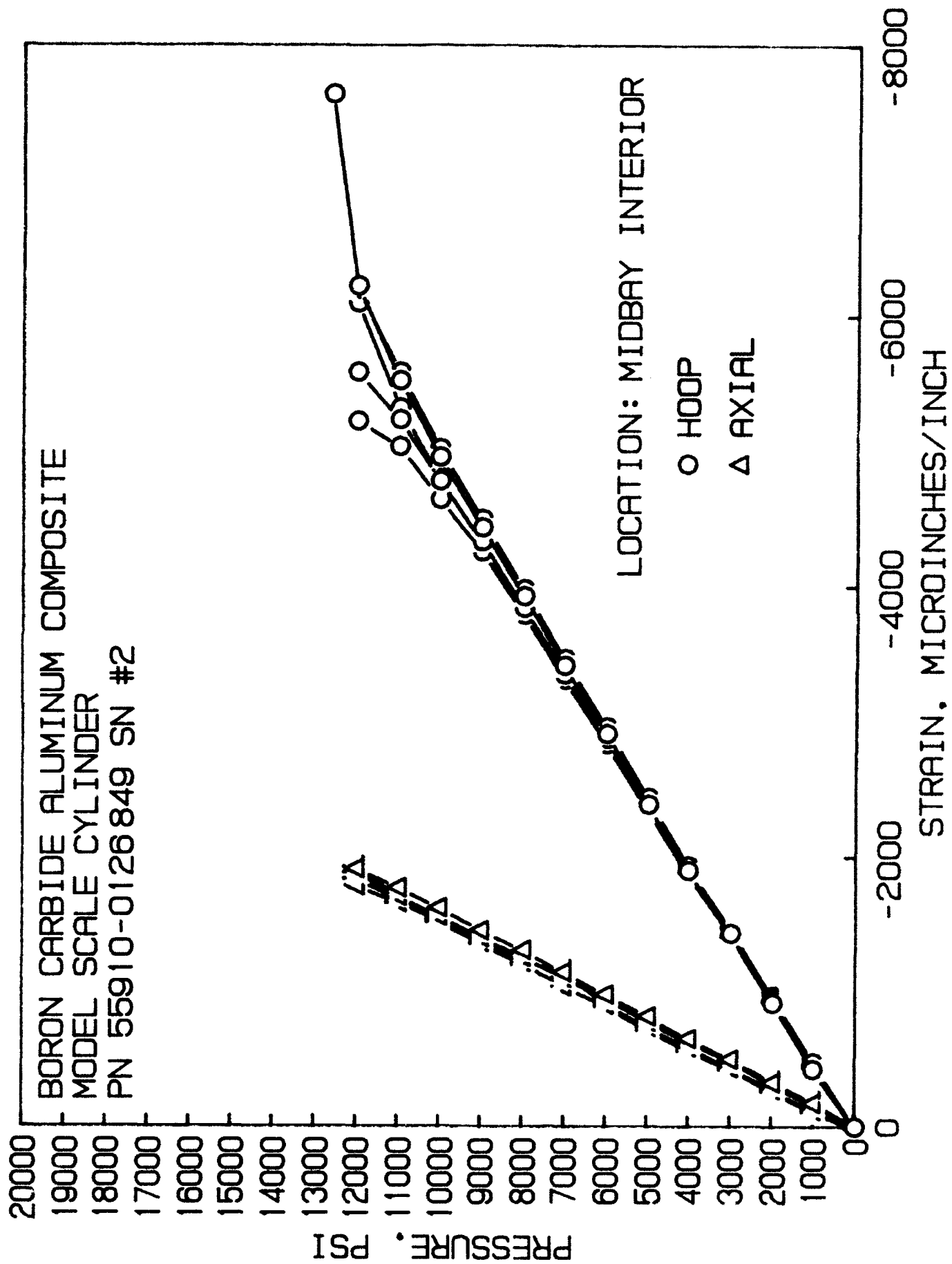
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-0126849 SN# 2

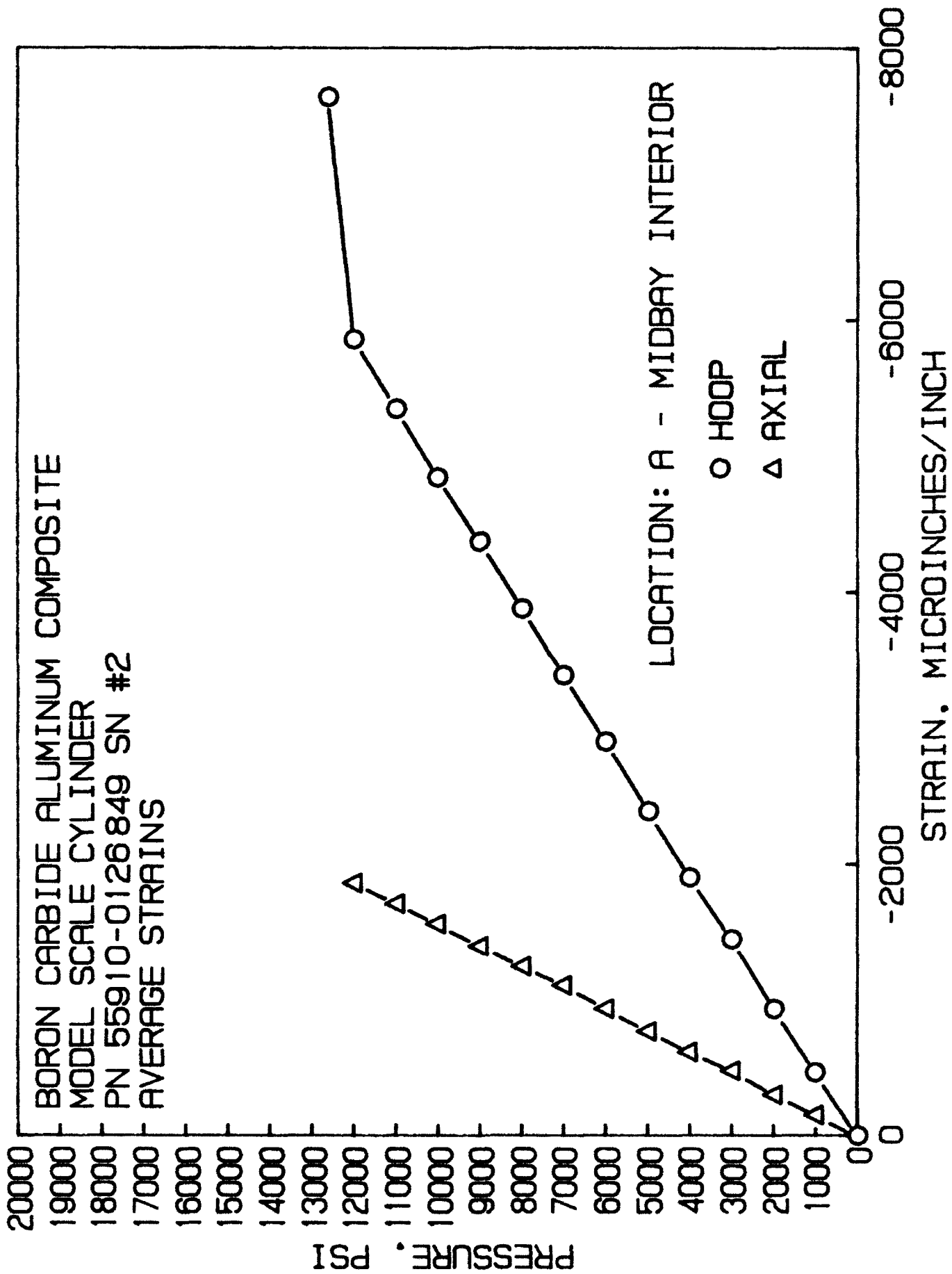
Pressurization # 1

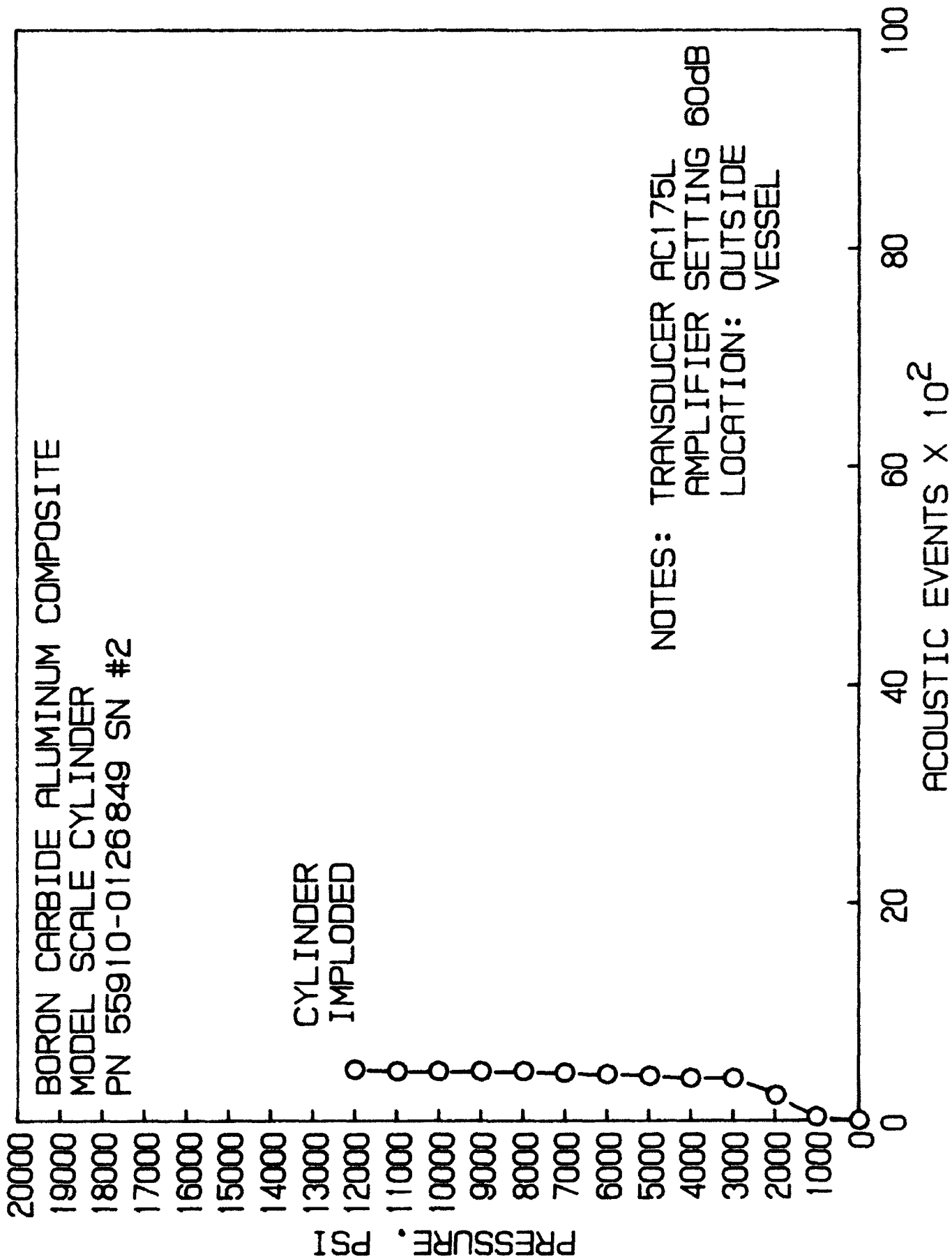
Pressure	Events	Time	Notes:
0000	0	9:07	1. Transducer: AET AC175
1000	25-25	9:10	SN# 7799 5 to 200 KHZ
2000	239-243	9:11	2. Amplifier Setting:
3000	385-386	9:13	Rate: T
4000	396-396	9:15	Gain: 60 DB
5000	414-416	9:17	Threshold: Automatic
6000	426-426	9:19	Function: Events
7000	440-440	9:21	3: Recorder:
8000	447-447	9:23	Channel "A" Events,
9000	458-458	9:25	4000 Full Range
10000	460-460	9:27	Channel "B" Rms,
11000	462-463	9:30	50 MV Full Scale,
12000	468-470	9:32	0.5 CM/Min Chart Scale

Failed at 12,600 psig









Test Cylinder SN#3 Type 3

Table 3 Strains on Dow Ceramic Cylinder PN 55910-01216849 SN# 3
Under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-538	-172	-479	-172	-515	-183	-517	-144	-501	-168
2000	-980	-322	-932	-327	-974	-335	-973	-293	-956	-321
3000	-1453	-473	-1415	-482	-1457	-487	-1456	-439	-1428	-474
4000	-1917	-623	-1893	-637	-1939	-640	-1935	-589	-1905	-630
5000	-2406	-786	-2391	-804	-2443	-803	-2436	-749	-2404	-791
6000	-2898	-960	-2911	-987	-2970	-982	-2959	-922	-2932	-966
7000	-3419	-1125	-3420	-1155	-3489	-1141	-3472	-1082	-3452	-1121
8000	-3864	-1273	-3877	-1307	-3960	-1284	-3943	-1228	-3927	-1264
9000	-4340	-1429	-4348	-1470	-4452	-1438	-4437	-1384	-4433	-1416
10000	-4783	-1586	-4776	-1632	-4921	-1586	-4916	-1537	-4922	-1561
11000	-5140	-1750	-5099	-1798	-5362	-1730	-5416	-1687	-5412	-1703
12000	-4638	-1966	-3946	-1986						

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-120, Gage Factor 2.11

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick

Cylinder Weight: 597 grams

Catastrophic Implosion 12,000 psi

Maximum compressive hoop stress at failure: 288,000 psi

Weight to Displacement Ratio: 0.25

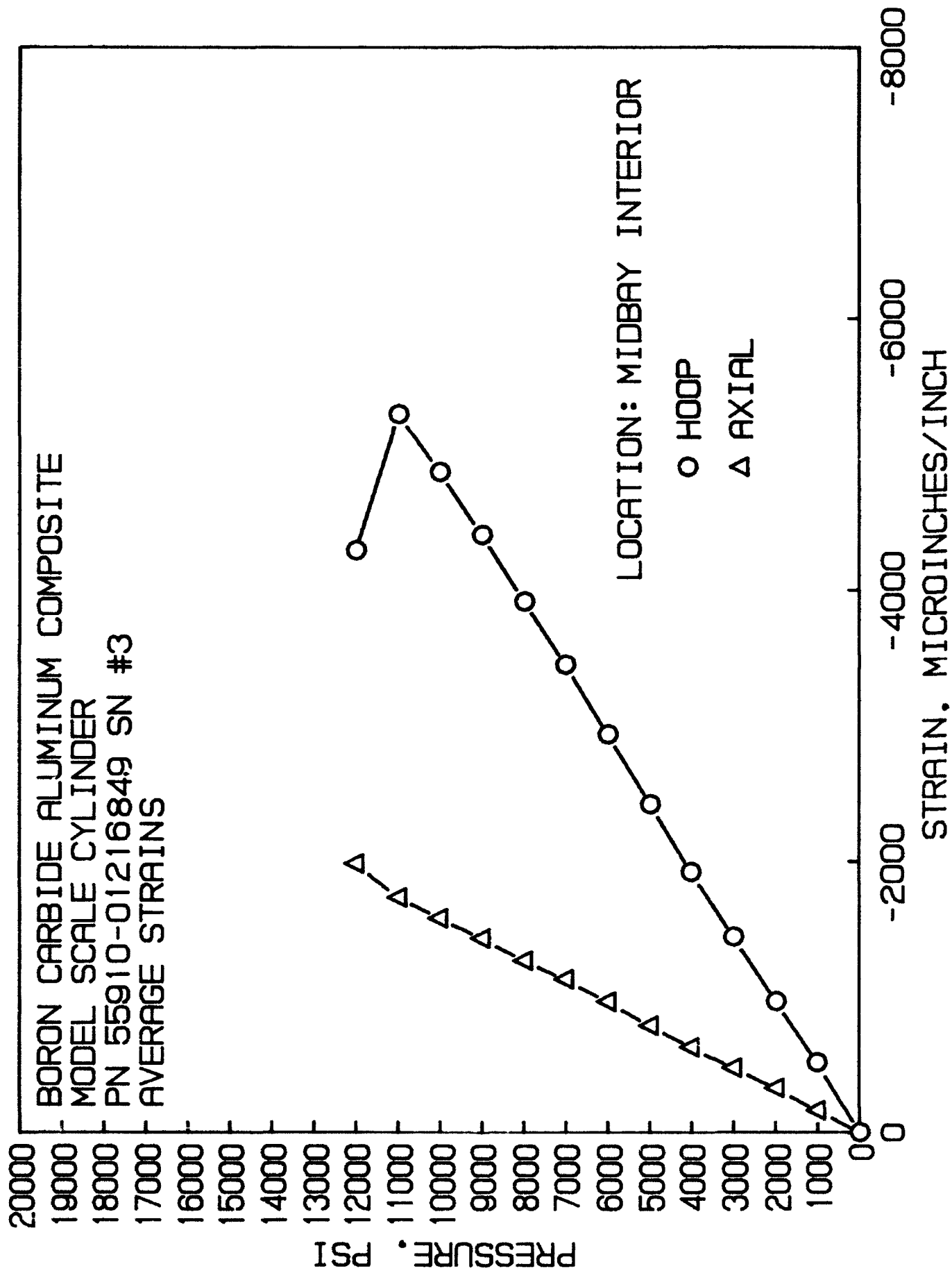
Test Date: 01-19-92

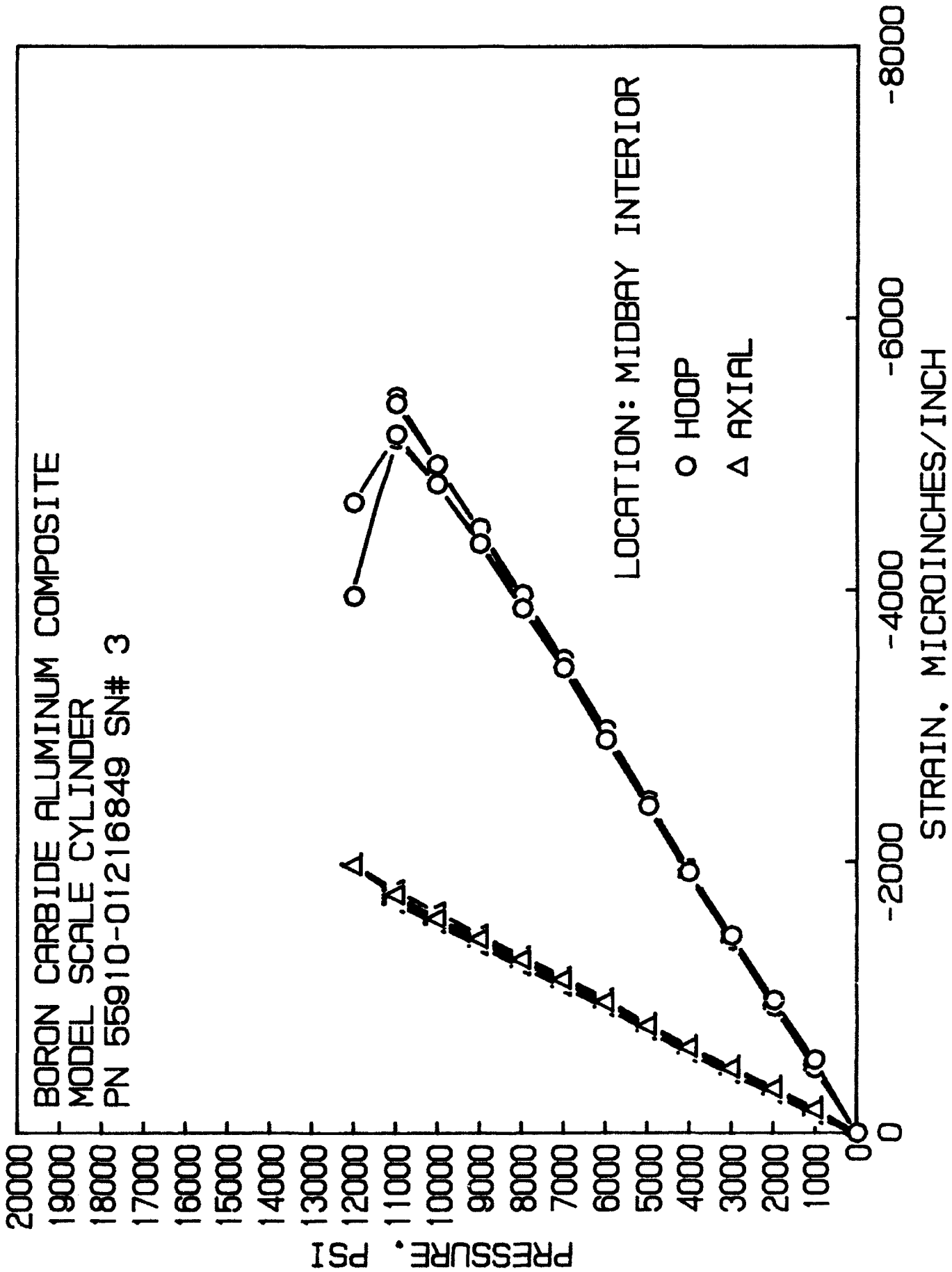
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-01216849 SN# 3

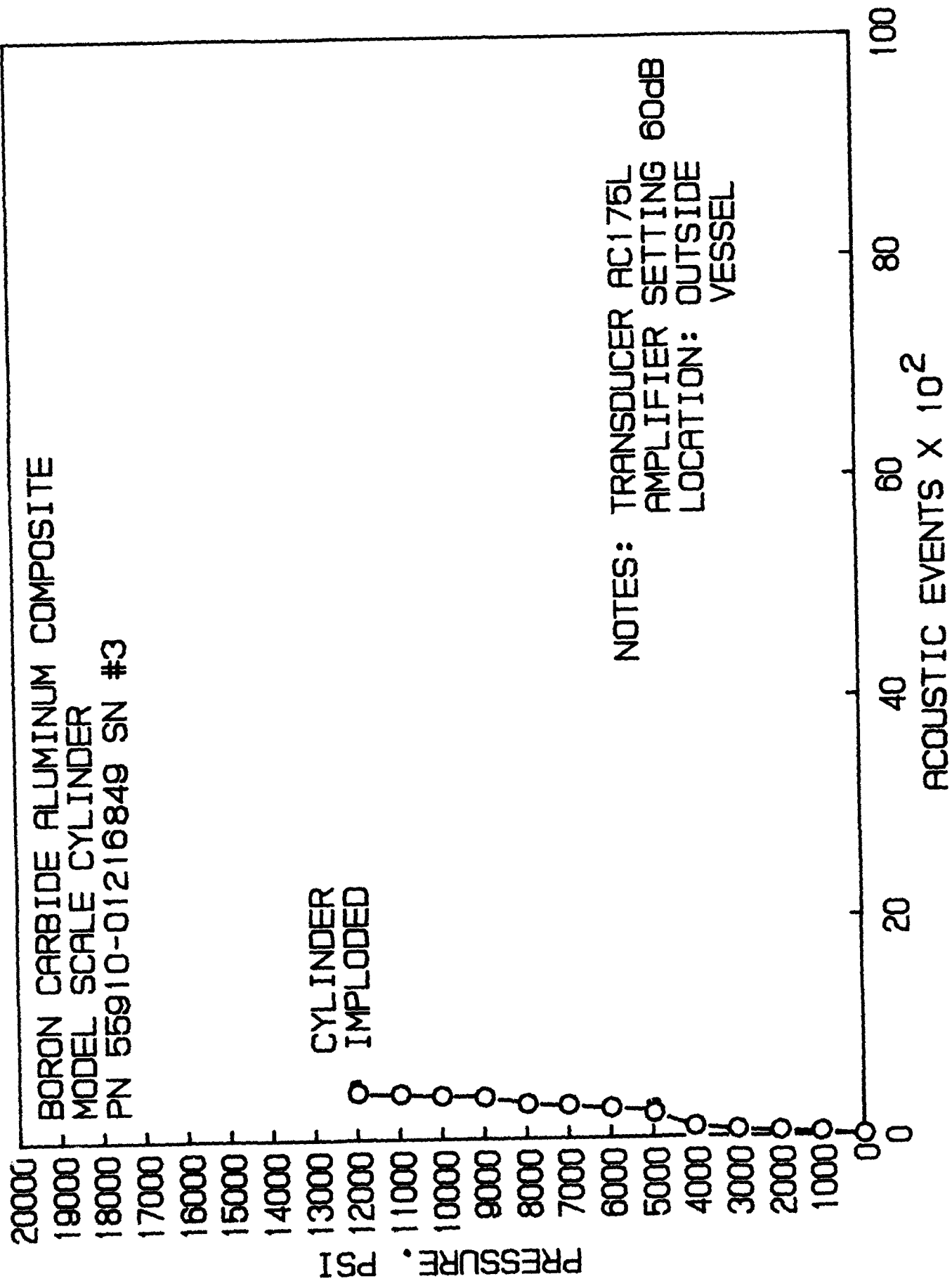
Pressurization # 1

Pressure	Events	Time	Notes:
0000	0	4:15	1. Transducer: AET AC175
1000	32-34	4:17	SN# 7799 5 to 200 KHZ
2000	50-53	4:20	2. Amplifier Setting:
3000	64-68	4:21	Rate: T
4000	90-96	4:23	Gain: 60 DB
5000	214-251	4:26	Threshold: Automatic
6000	264-272	4:29	Function: Events
7000	291-299	4:30	3: Recorder:
8000	310-322	4:31	Channel "A" Events,
9000	382-392	4:32	4000 Full Range
10000	397-398	4:33	Channel "B" Rms,
11000	409-413	4:34	50 MV Full Scale,
12000	423-456	4:35	0.5 CM/Min Chart Scale

Failed at 12,000 psig







Test Cylinder SN#4 Type 3

Table 4 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 4
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location A - Midway									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-454	-148	-531	-189	-471	-153	-509	-157	-528	-174
2000	-954	-296	-1035	-332	-981	-296	-1036	-296	-1046	-319
3000	-1478	-460	-1560	-485	-1517	-452	-1585	-450	-1583	-473
4000	-1970	-613	-2057	-627	-2020	-599	-2101	-596	-2090	-617
5000	-2486	-775	-2581	-771	-2546	-751	-2642	-747	-2620	-766
6000	-3053	-951	-3150	-932	-3118	-916	-3233	-910	-3204	-930
7000	-3575	-1110	-3682	-1074	-3650	-1067	-3776	-1059	-3738	-1076
8000	-4143	-1282	-4259	-1228	-4229	-1226	-4366	-1220	-4319	-1235
9000	-1444	-48	-4782	-1374	-4782	-1374	-4865	-1369	-4865	-1382
0	-48	-607	-270	-21	-270	-21	-27	-295	-295	-29
4000	-1255	-1588	-2329	-613	-2329	-613	-1927	-614	-1938	-592
8000	-1738	-1776	-4467	-1234	-4467	-1234	-4099	-1232	-4077	-1205
10000			-5555	-1541	-5555	-1541	-5160	-1550	-5156	-1515
11000			-6104	-1689	-6104	-1689	-5540	-1721	-5659	-1674
11500			-6365	-1758	-6365	-1758	-5262	-1857	-5741	-1764
11800							-4037	-1927		

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick

Cylinder Weight: 584 grams

Catastrophic implosion occurred 11,800 psi

Maximum compressive hoop stress at test termination: 283,000 psi

Test Date: 03-17-92

ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-01216849 SN# 4

Pressurization # 1

Pressure	Events	Time	Notes:
0000	0	4:12	1. Transducer: AET AC175
1000	8-8	4:15	SN# 7799 5 to 200 KHZ
2000	65-72	4:17	2. Amplifier Setting:
3000	127-132	4:18	Rate: T
4000	172-180	4:20	Gain: 60 DB
5000	204-206	4:22	Threshold: Automatic
6000	223-226	4:24	Function: Events
7000	232-233	4:25	3. Recorder:
8000	238-238	4:29	Channel "A" Events,
9000	249-258	4:30	4000 Full Range
			Channel "B" Rms,
			50 MV Full Scale,
			0.5 CM/Min Chart Scale

Test terminated at 9,000 psig without failure

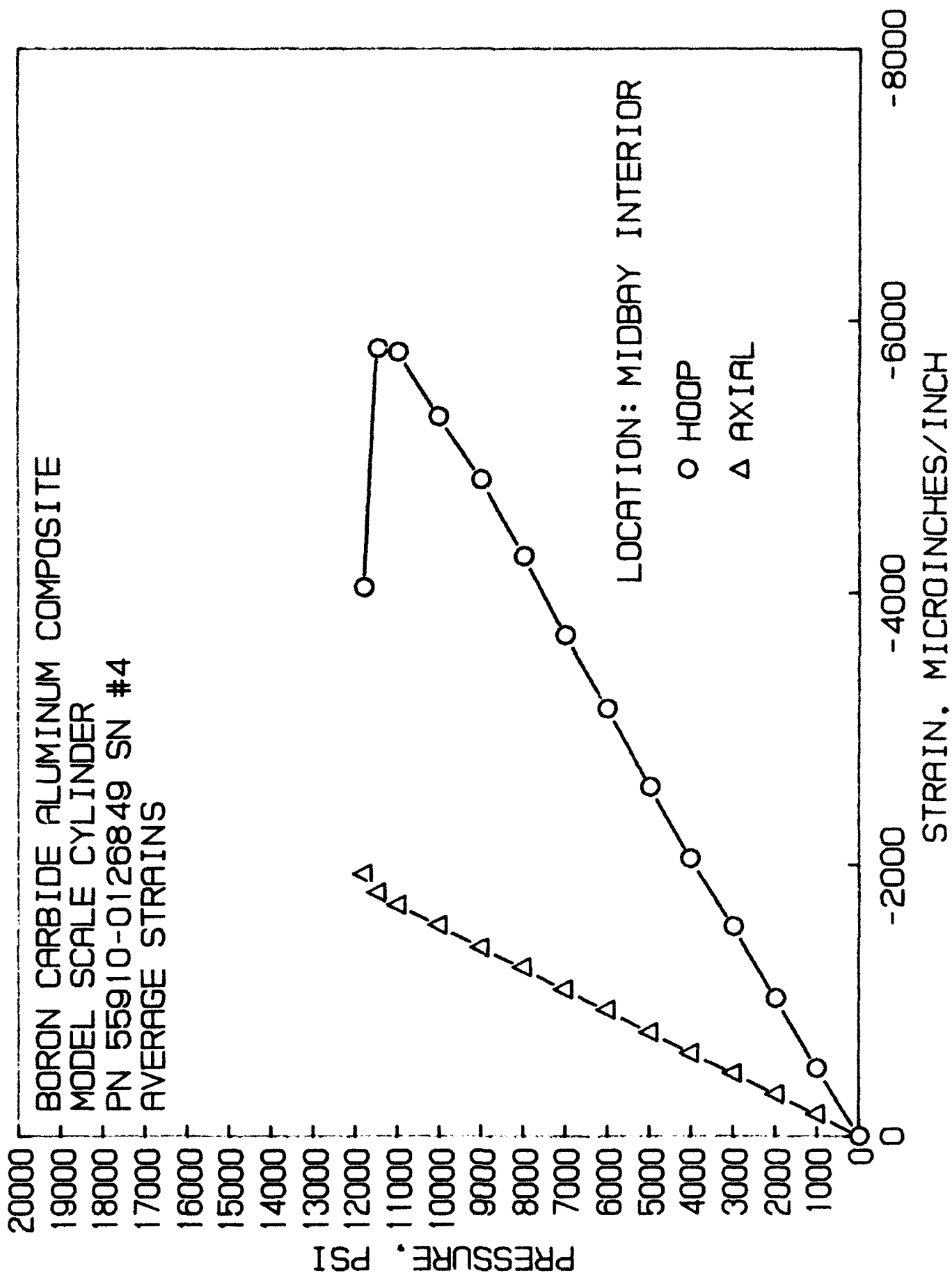
Test Date: 03-17-92

ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-01216849 SN# 4

Pressurization # 2

Pressure	Events	Time	Notes:
0000	0	6:45	1. Transducer: AET AC175
1000	21-22	6:47	SN# 7799 5 to 200 KHZ
2000	32-32	6:49	2. Amplifier Setting:
3000	38-38	6:50	Rate: T
4000	50-50	6:52	Gain: 60 DB
5000	69-69	6:53	Threshold: Automatic
6000	78-83	6:54	Function: Events
7000	89-89	6:55	3. Recorder:
8000	94-94	6:57	Channel "A" Events,
9000	100-100	6:58	4000 Full Range
10000	110-110	7:00	Channel "B" Rms,
10500	111-112	7:02	50 MV Full Scale,
11000	113-114	7:03	0.5 CM/Min Chart Speed
11500	118-119	7:04	

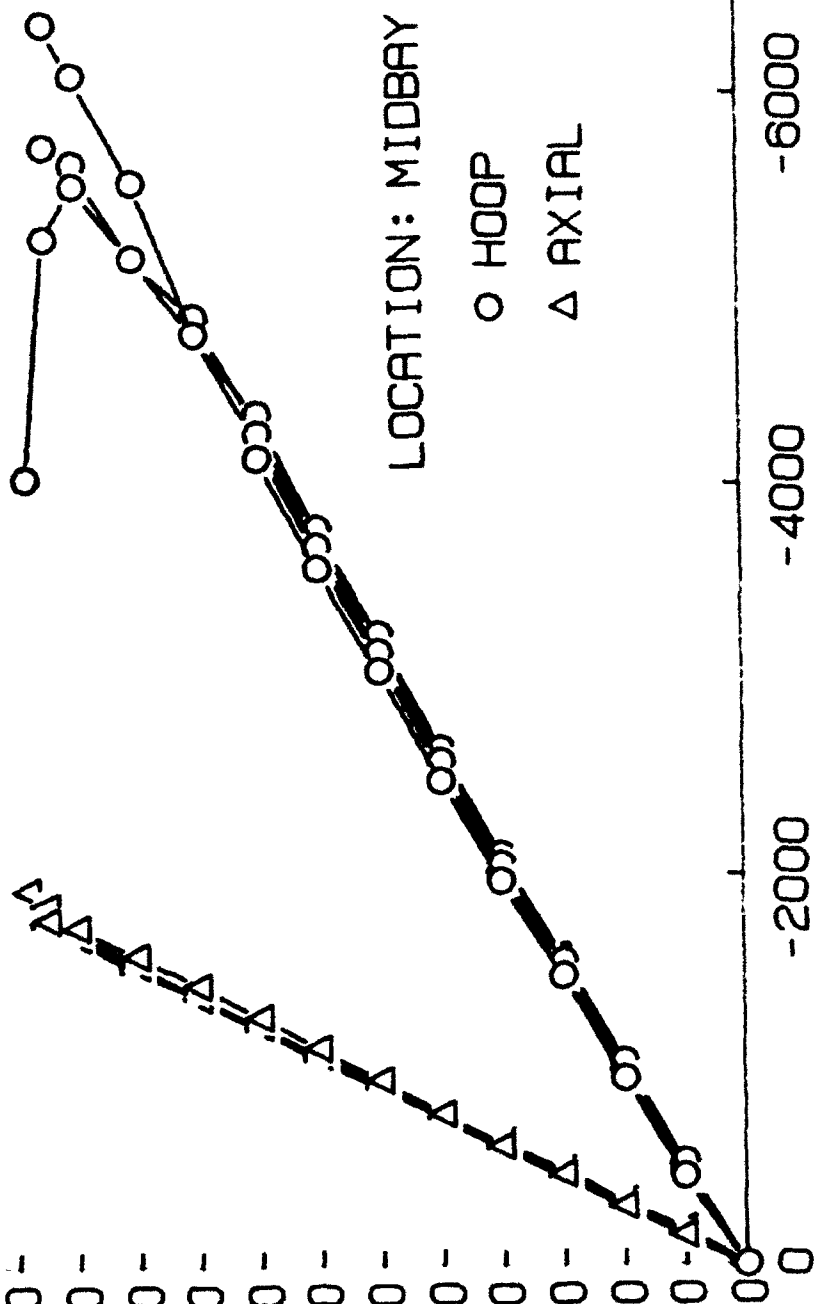
Failed at 11,800 psig

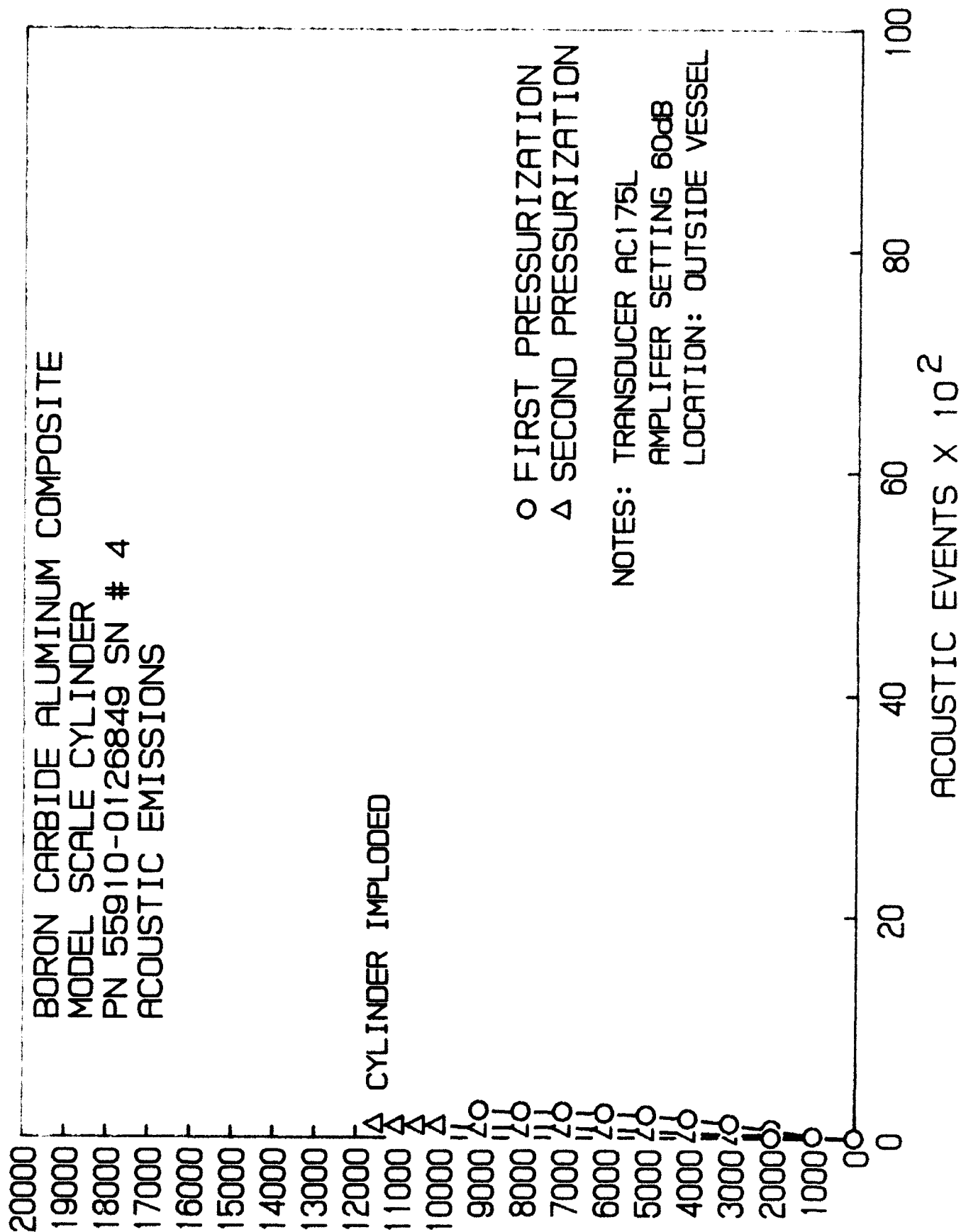


BORON CARBIDE ALUMINUM COMPOSITE
 MODEL SCALE CYLINDER
 PN 55910-0126849 SN #4
 STRAINS

PRESSURE, PSI

STRAIN, MICROINCHES/INCH
 LOCATION: MIDBAY INTERIOR
 O HOOP
 Δ AXIAL





Test Cylinder SN#5 Type 3

• Table 5. Tested with titanium hemispherical bulkheads

• Table 5.1. Tested with plane steel bulkheads

Table 5 Strains on Dow Ceramic Cylinder PN 55970-0126849 SN# 5
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location A - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-455	-136	-417	-160	-501	-196	-437	-139	-479	-183
2000	-892	-285	-863	-316	-949	-354	-911	-303	-936	-337
3000	-1316	-430	-1291	-463	-1393	-506	-1359	-465	-1366	-486
4000	-1743	-585	-1718	-622	-1838	-665	-1810	-634	-1810	-642
5000	-2163	-736	-2148	-776	-2279	-821	-2257	-800	-2246	-792
6000	-2607	-896	-2599	-938	-2744	-983	-2726	-975	-2704	-951
7000	-3060	-1068	-3060	-1111	-3211	-1156	-3199	-1161	-3169	-1122
8000	-3484	-1219	-3493	-1262	-3650	-1308	-3640	-1324	-3605	-1268
9000	-3926	-1378	-3950	-1421	-4104	-1472	-4095	-1502	-4065	-1423
10000	-4356	-1538	-4399	-1577	-4546	-1632	-4529	-1681	-4513	-1577
10500	-4559	-1616	-4614	-1649	-4757	-1705	-4728	-1765	-4724	-1648
11000	-4808	-1704	-4876	-1735	-4980	-1795	-4943	-1866	-4974	-1730
11500	-5030	-1781	-5114	-1803	-5167	-1873	-5094	-1961	-5182	-1798
12000	-5262	-1851	-5354	-1865	-5300	-1947	-5162	-2054	-5356	-1862
12500	-5600	-1914	-5704	-1912	-5280	-2042	-4848	-2194	-5433	-1925

NOTES: ALL strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 4.956 L x 0.125 in thick

Cylinder Weight: 562 grams

Test terminated without failure at 12,500 psi

Maximum compressive hoop stress at test termination: 300,000 psi

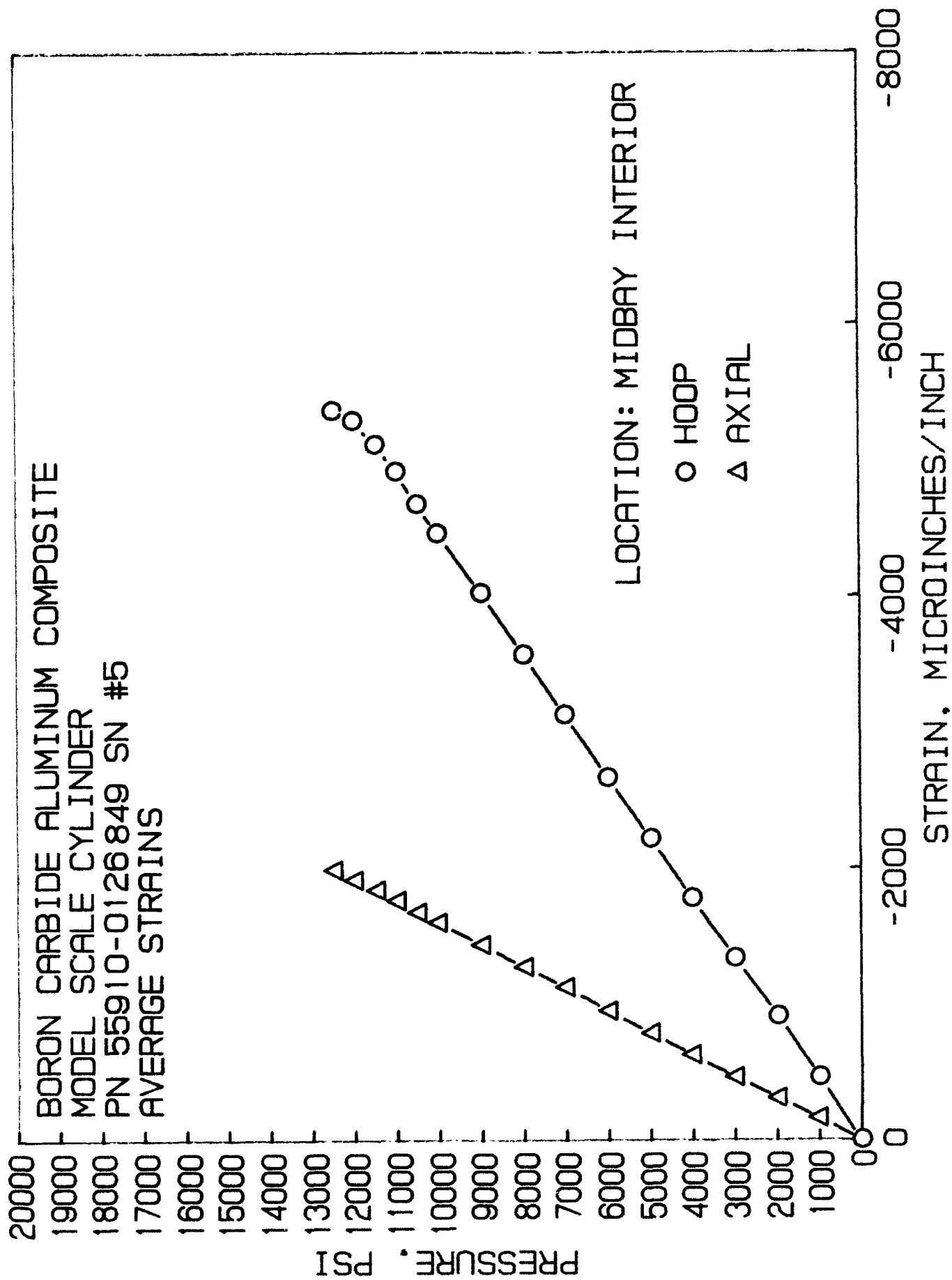
Test Date: 03-15-92

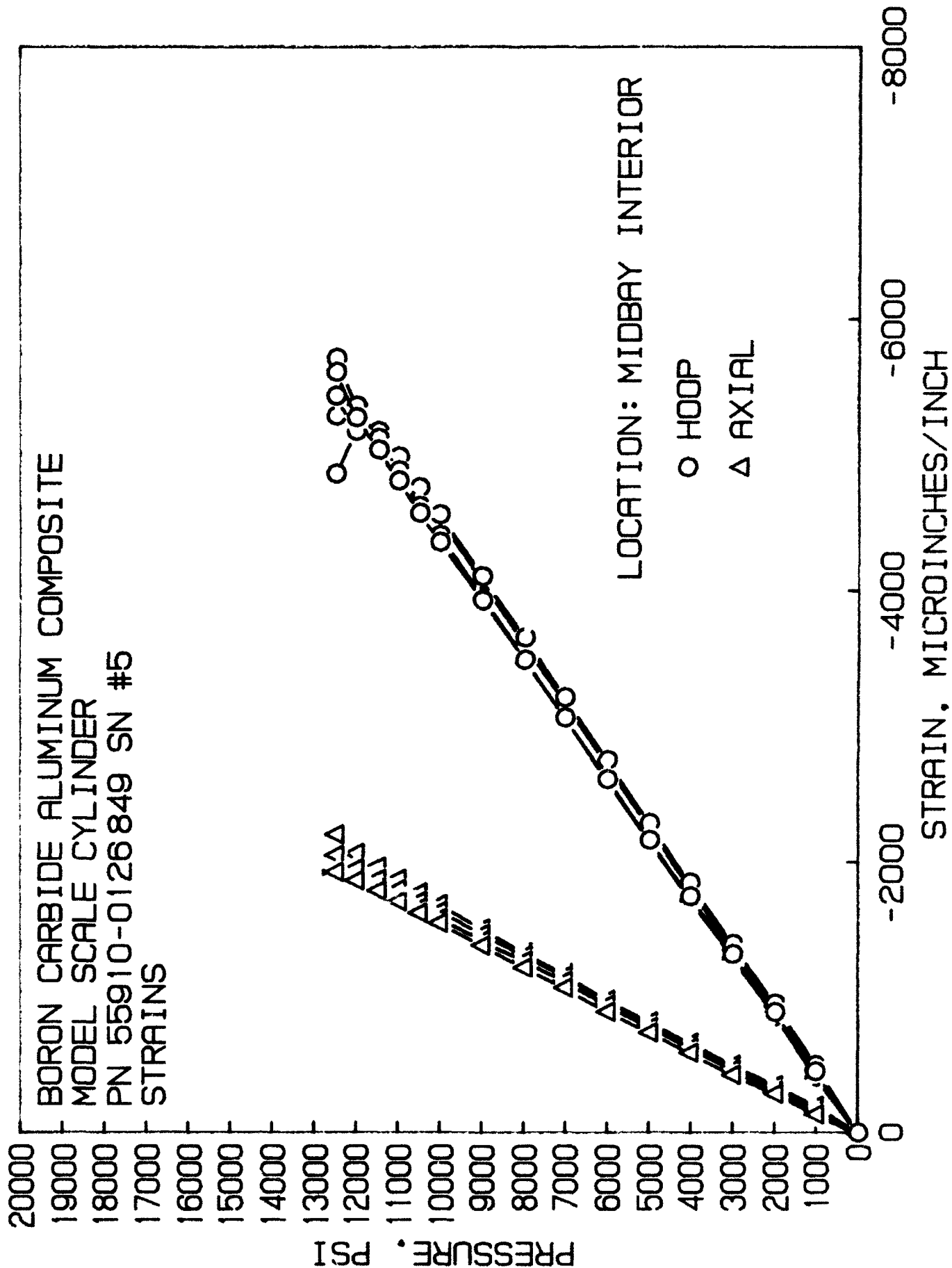
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-01216849 SN# 5

Pressurization # 1

Pressure	Events	Time	Notes:
0000	0	2:10	1. Transducer: AET AC175
1000	27-30	2:13	SN# 7799 5 to 200 KHZ
2000	95-107	2:16	2. Amplifier Setting:
3000	135-163	2:19	Rate: T
4000	177-224	2:20	Gain: 60 DB
5000	231-231	2:22	Threshold: Automatic
6000	251-257	2:23	Function: Events
7000	275-276	2:26	3: Recorder:
8000	281-281	2:30	Channel "A" Events,
9000	290-292	2:31	4000 Full Range
10000	295-297	2:33	Channel "B" Rms,
10500	306-307	2:35	50 MV Full Scale,
11000	310-310	2:37	0.5 CM/Min Chart Scale
11500	319-319	2:38	
12000	321-322	2:40	
12500	325-328	2:41	

Test terminated at 12,500 psig without implosion





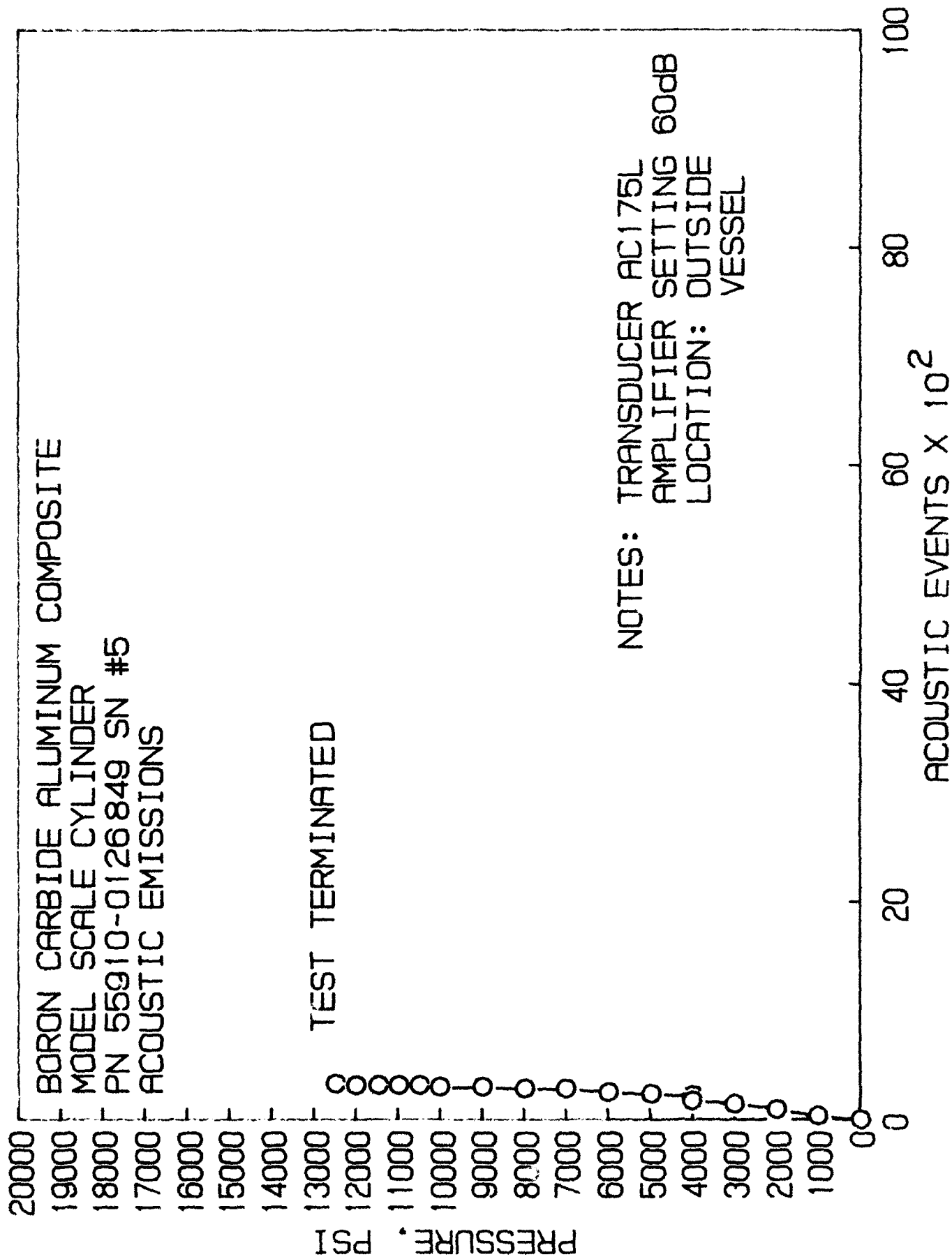


Table 5-1 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 5
under Short Term Pressurizations with Ends Radially Supported by Plane Steel Bulkheads

Interior Gage Locations

Pressure (Psi)	Location A - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
2000	-908	-425	-842	-257	-859	-278	-814	-418	-972	-163
4000	-1844	-763	-1696	-572	-1742	-582	-1715	-749	-1890	-479
6000	-2748	-1094	-2538	-892	-2612	-888	-2589	-1071	-2795	-806
8000	-3698	-1448	-3433	-1231	-3518	-1216	-3496	-1414	-3760	-1149
10000	-4579	-1779	-4277	-1553	-4370	-1522	-4342	-1735	-4659	-1473
12000	-5490	-2124	-5158	-1883	-5273	-1837	-5220	-2056	-5622	-1904
12200	-5575	-2150	-5241	-1909	-5359	-1869	-5307	-2079	-5724	-1832
12400	-5667	-2182	-5326	-1939	-5449	-1899	-5396	-2106	-5825	-1860
12600	-5769	-2217	-5423	-1976	-5547	-1936	-5498	-2137	-5941	-1898
12800	-5874	-2257	-5522	-2017	-5647	-1976	-5601	-2176	-6059	-1936
13000	-5984	-2295	-5625	-2057	-5746	-2018	-5711	-2211	-6187	-1975
13200	-6067	-2325	-5701	-2085	-5825	-2048	-5791	-2236	-6285	-2002
13400	-6165	-2357	-5790	-2119	-5911	-2082	-5893	-2259	-6395	-2031
13600	-6264	-2387	-5874	-2150	-6000	-2113	-5991	-2293	-6519	-2067
13800	-6371	-2424	-5966	-2185	-6091	-2154	-6095	-2322	-6647	-2099
14000	-6489	-2460	-6066	-2221	-6191	-2192	-6213	-2355	-6790	-2136
14200	-6589	-2488	-6153	-2252	-6273	-2226	-6317	-2383	-6916	-2168
14400	-6715	-2526	-6266	-2288	-6384	-2265	-6446	-2419	-7079	-2206
14600	-6864	-2565	-6381	-2327	-6507	-2306	-6604	-2442	-7263	-2234
14800	-7008	-2585	-6485	-2350	-6611	-2334	-6751	-2475	-7462	-2270
15000	-7196	-2624	-6639	-2390	-6775	-2374	-6986	-2512	-7751	-2305
15200	-7423	-2645	-6803	-2414	-6949	-2409	-7250	-2541	-8091	-2327
15400	-7800	-2574	-7094	-2454	-7266	-2454	-7766	-2582	-8746	-2355
15600	-8957									

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Plane Steel Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 4.956 L x 0.125 in thick

Cylinder Weight: 562 grams

Cylinder Imploded at 15,600 psi

Maximum compressive hoop stress at cylinder implosion: 374,400 psi

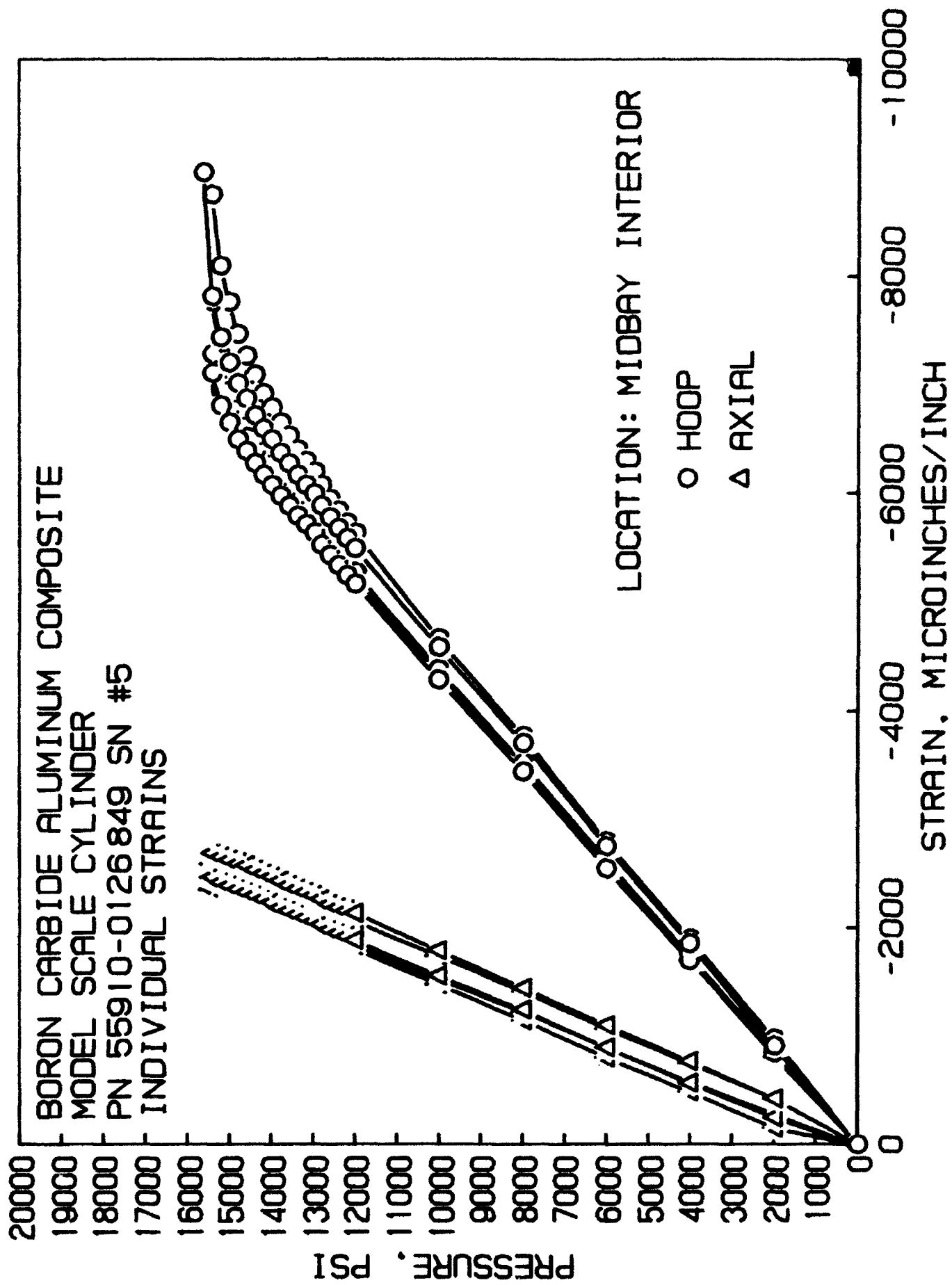
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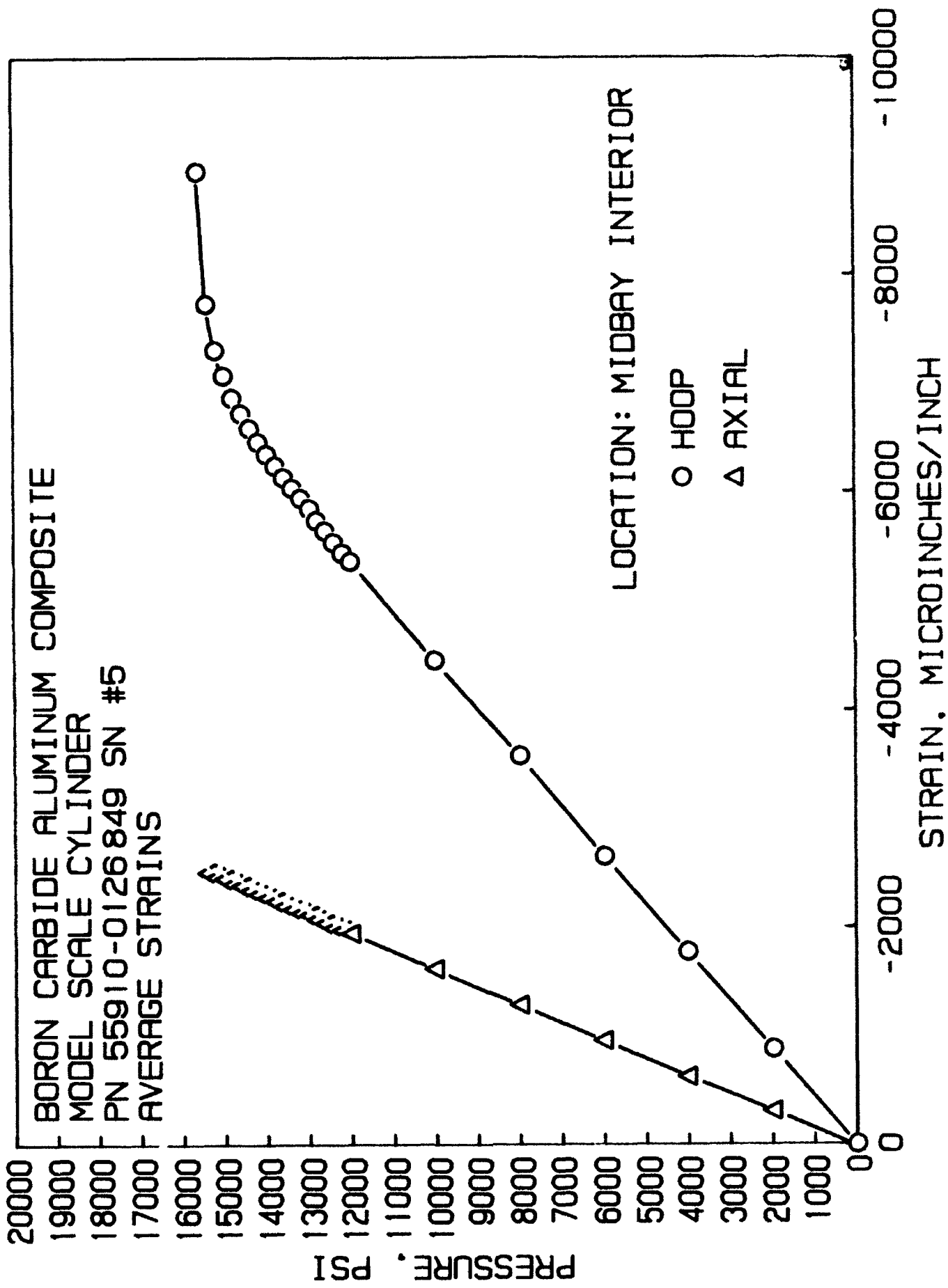
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-01216847 SN# 05

Pressurization # 1

Pressure	Events	Time	Notes:
0000	0	5:10	1. Transducer: AET AC175
1000	3-3	5:13	SN# 7799 5 to 200 KHZ
2000	5-5	5:15	2. Amplifier Setting:
3000	18-20	5:16	Rate: T
4000	21-22	5:18	Gain: 60 DB
5000	24-24	5:20	Threshold: Automatic
6000	24-25	5:21	Function: Events
7000	27-29	5:23	3. Recorder:
8000	63-63	5:25	Channel "A" Events,
9000	67-69	5:26	4000 Full Range
10000	75-75	5:30	Channel "B" Rms,
11000	80-90	5:32	50 MV Full Scale,
12000	130-164	5:33	0.5 CM/Min Chart Scale
12500	172-172	5:35	
13000	175-181	5:37	
13200	196-196	5:40	
13400	196-200	5:41	
13500	212-215	5:43	

Failed at 13,500 psig





Test Cylinder SN#6 Type 3

Table 6. Tested with titanium hemispherical bulkheads

Table 6.1. Tested with plane steel bulkheads

Table 6 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 6
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-459	-132	-487	-167	-471	-181	-477	-141	-499	-171
2000	-919	-283	-969	-324	-946	-340	-957	-297	-964	-321
3000	-1399	-438	-1465	-486	-1432	-504	-1456	-458	-1442	-473
4000	-1829	-577	-1913	-631	-1871	-650	-1908	-601	-1875	-612
5000	-2310	-733	-2407	-794	-2362	-812	-2411	-761	-2352	-765
6000	-2772	-885	-2890	-951	-2840	-969	-2898	-916	-2815	-914
7000	-3225	-1032	-3348	-1103	-3302	-1117	-3366	-1065	-3256	-1058
8000	-3695	-1189	-3849	-1267	-3803	-1280	-3873	-1226	-3738	-1212
9000	-4145	-1340	-4323	-1423	-4279	-1432	-4355	-1379	-4193	-1356
10000	-4591	-1487	-4798	-1577	-4763	-1581	-4838	-1531	-4647	-1497
11000	-5072	-1652	-5322	-1748	-5304	-1742	-5370	-1699	-5136	-1654
12000	-5467	-1802	-5804	-1897	-5830	-1878	-5847	-1852	-5543	-1789
13000	-5746	-1975	-6416	-2043	-6628	-1989	-6418	-2013	-5820	-1942
10000	-4589	-1498	-4844	-1584	-4825	-1577	-4874	-1537	-4652	-1502
0	-51	-16	-20	-12	-19	-12	-19	-10	-40	-8
10000	-4574	-1478	-4820	-1569	-4772	-1567	-4850	-1526	-4626	-1490
13000	-5701	-1961	-6443	-2027	-6656	-1971	-6413	-2007	-5773	-1930
13300	-5562									
0	-21	-5	-11	-8	-9	-3	-10	-4	-12	-2

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick

Cylinder Weight: 597 grams

Test terminated without implosion at 13,300 psi

Maximum compressive hoop stress at test termination: 319,200 psi

Test Date: 5-1-92

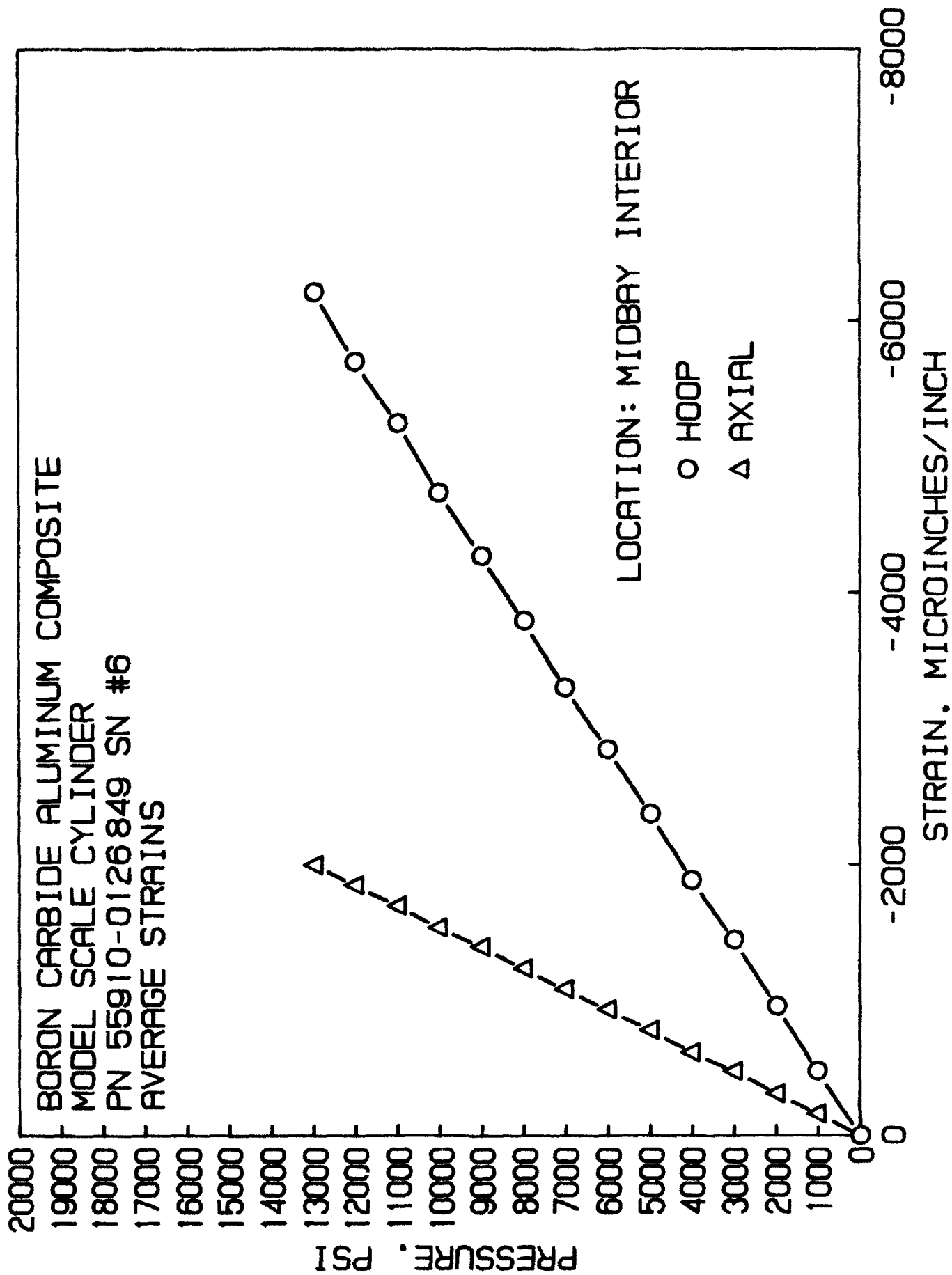
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER 55910-0126849 SN# 06

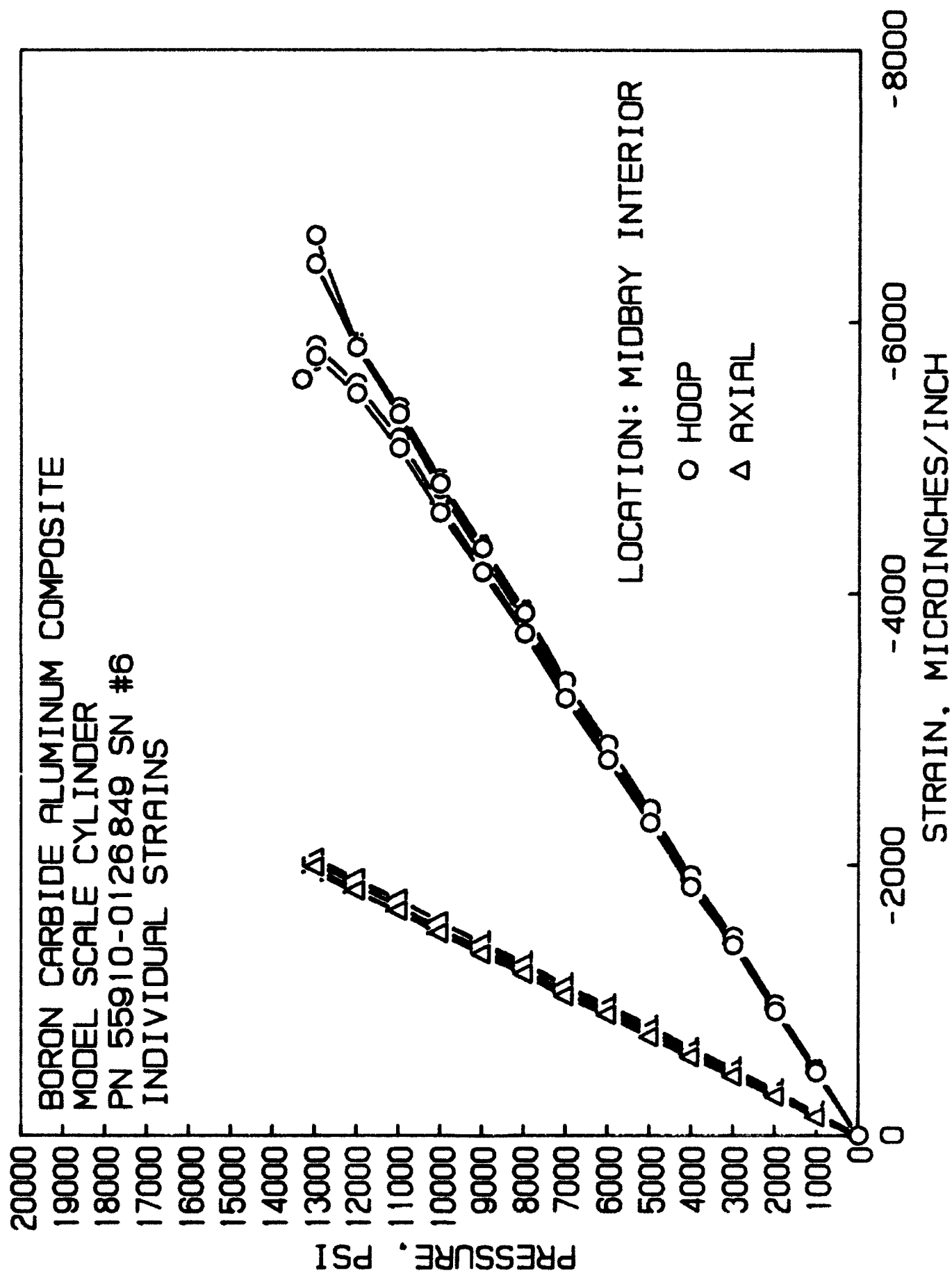
Pressurizations 1 and 2

Pressure	Events	
	Pressurization 1	2
0000	0	0
1000	5	2
2000	21	2
3000	70	3
4000	115	3
5000	120	3
6000	123	3
7000	124	3
8000	127	3
9000	138	3
10000	139	3
11000	144	3
12000	145	3
13000	149	3
13300	179	4

Test terminated at 13,300 psi due to impending catastrophic failure.

- Notes: 1. Transducer: AET AC175 SN#7799
5 to 200 KHZ
2. Amplifier Setting:
Rate: T Gain: 60 DB
 Threshold: Automatic
 Function: Events
3. Recorder:
Channel "A" Events,
400 Full Range,
Channel "B" Rms,
50 MV Full Scale,
0.5 CM/Min Chart Speed





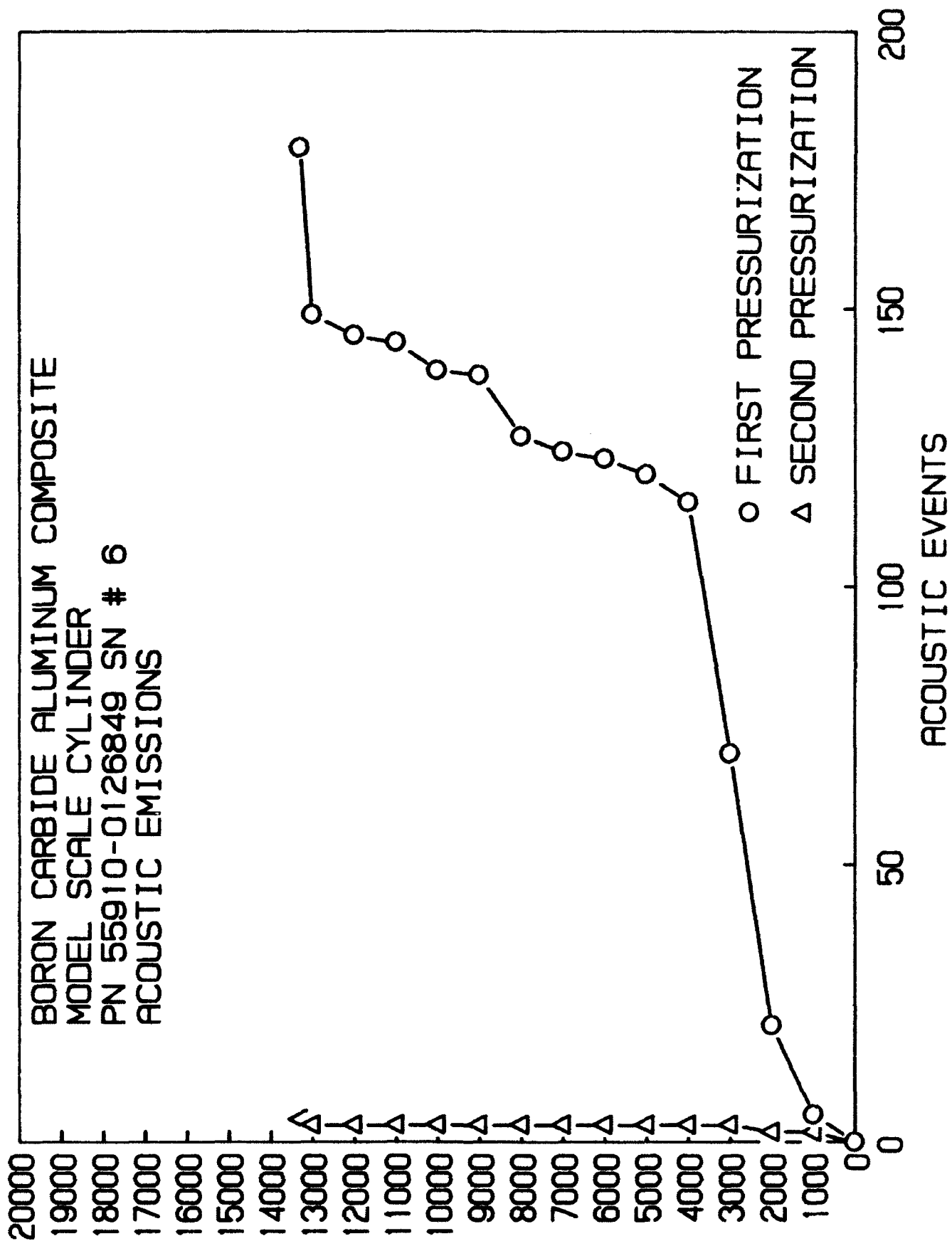


Table 6-1 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 6
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location A - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
2000	-1090	-264	-980	-471	-1000	-380	-1122	-321	-946	-464
4000	-2010	-550	-1922	-785	-1907	-713	-2089	-613	-1841	-777
6000	-2925	-846	-2871	-1097	-2830	-1035	-3084	-908	-2760	-1087
8000	-3860	-1147	-3819	-1421	-3762	-1364	-4092	-1211	-3697	-1399
10000	-4800	-1436	-4777	-1738	-4676	-1672	-5096	-1508	-4673	-1712
12000	-5693	-1718	-5704	-2051	-5622	-1972	-6095	-1803	-5586	-2008
13000	-6131	-1865	-6152	-2211	-6083	-2131	-6597	-1954	-6019	-2142
14000	-6626	-2024	-6668	-2384	-6604	-2303	-7168	-2123	-6521	-2307
15000	-7134	-2183	-7207	-2555	-7165	-2473	-7784	-2291	-7040	-2461
15500									-7389	-2532

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125UT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

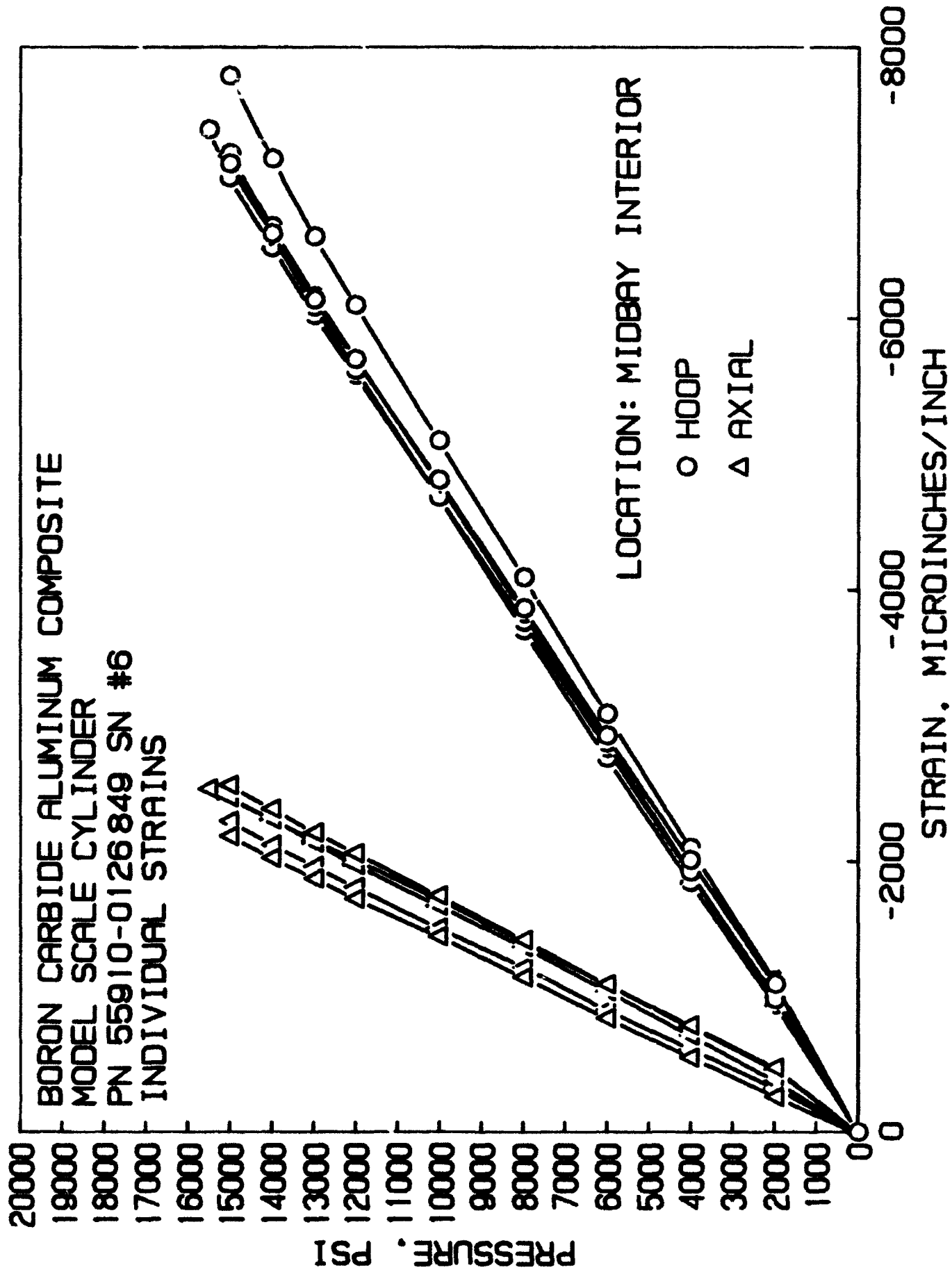
End Closures: Plane Steel Bulkheads providing radial support

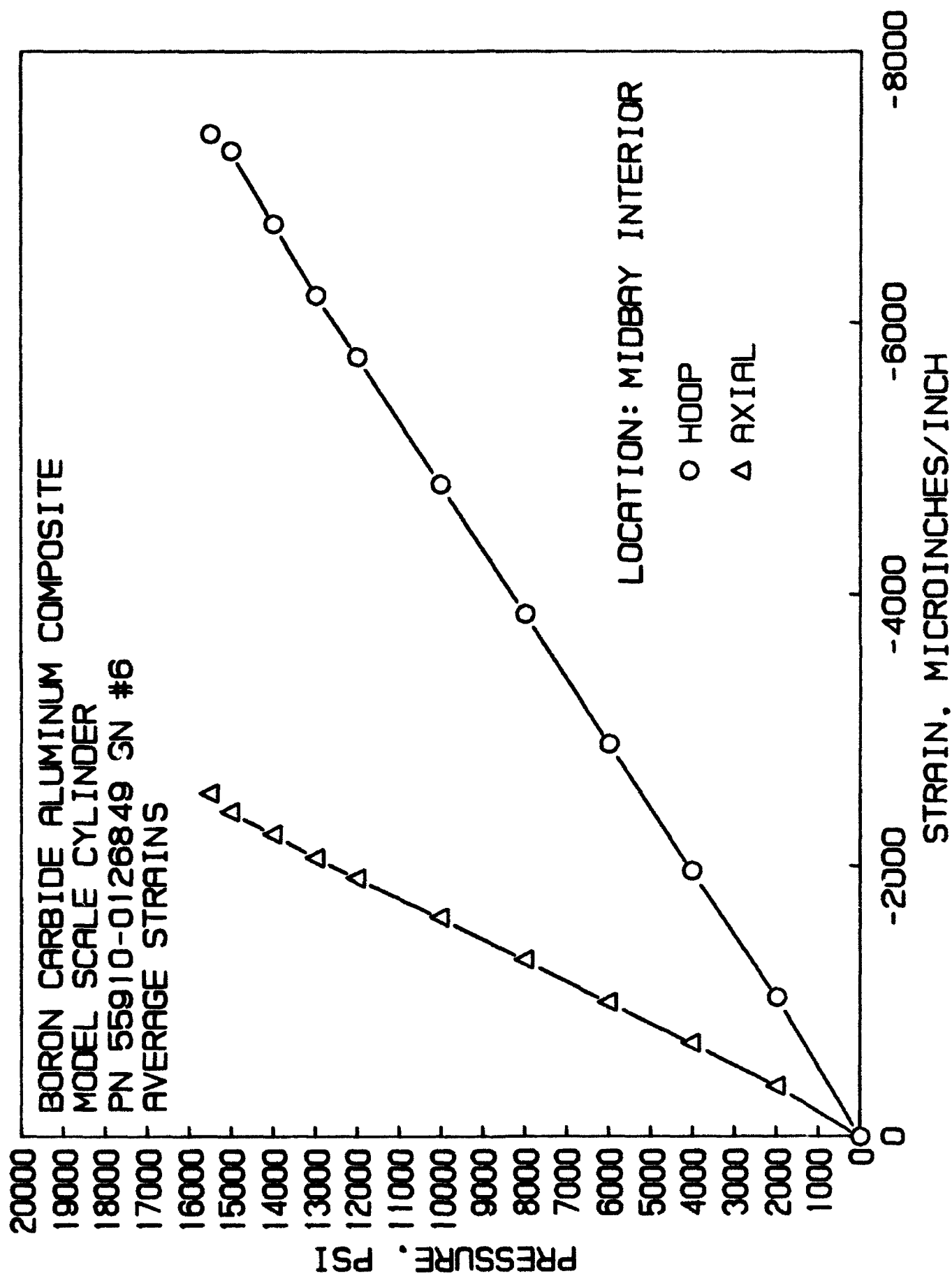
Cylinder Dimensions: 6.000 OD x 5.000 L x 0.125 in thick

Cylinder Weight: 597 grams

Catastrophic implosion occurred at 15,500 psi

Maximum compressive hoop stress at implosion: 372,000 psi





Test Cylinder SN#7 Type 1

Table 7 Strains on Dow Ceramic Cylinder PN 55910-0126847 SM# 7
under Short Term Pressurizations

Interior Gage Locations

Location A - Midbay

Pressure (Psi)	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-335	-109	-317	-139	-352	-191	-265	90	-45	147
2000	-492	-135	-633	-208	-596	-384	-599	-251	-618	-244
3000	-922	-291	-903	-353	-960	-526	-967	-364	-964	-381
4000	-1249	-439	-1220	-475	-1287	-640	-1282	-478	-1286	-499
5000	-1545	-543	-1508	-582	-1576	-742	-1565	-581	-1581	-608
6000	-1845	-648	-1801	-689	-1884	-844	-1861	-689	-1883	-712
7000	-2137	-753	-2105	-797	-2196	-946	-2163	-799	-2194	-816
8000	-2450	-849	-2402	-904	-2503	-1047	-2461	-908	-2503	-918
9000	-2747	-955	-2693	-1014	-2810	-1147	-2755	-1016	-2796	-1022
10000	-3035	-1053	-2985	-1122	-3111	-1244	-3046	-1118	-3111	-1126
0	-39	-64	-74	-59	-108	-237	-121	-71	-109	-79
10000	-2957	-1013	-2910	-1063	-3019	-1008	-2928	-1060	-3029	-1056
0	-13	-26	-6	-1	-3	-11	-10	-16	-24	-12
10000	-2970	-1012	-2907	-1071	-2989	-990	-2929	-1067	-3010	-1045
0	1	-18	-7	-8	-8	-7	-4	-10	-22	-10
10000	-3019	-1032	-2960	-1104	-3058	-1040	-2953	-1092	-3048	-1087
0	-55	-42	-48	-39	-40	-36	-33	-38	-40	-39
10000	-2968	-991	-2910	-1068	-3017	-1013	-2918	-1055	-3006	-1049
0	-13	-7	-10	-8	-8	-8	-9	-7	-4	-2
10000	-2963	-982	-2901	-1060	-3011	-991	-2911	-1050	-2996	-1040
0	7	10	8	8	10	12	9	7	11	12
10000	-2968	-990	-2904	-1066	-3016	-1000	-2913	-1056	-3000	-1047
0	-7	-1	-1	0	1	5	-1	2	5	7
10000	-2979	-992	-2914	-1067	-3028	-1002	-2923	-1056	-3012	-1048
0	-8	-6	-6	-4	-3	2	-3	-4	-1	2
10000	-2968	-990	-2905	-1066	-3117	-998	-2914	-1055	-3003	-1044
0	-3	-1	0	3	1	15	11	10	11	15
10000	-2968	-983	-2904	-1060	-3019	-990	-2911	-1047	-3002	-1037
0	-1	4	3	5	4	13	5	7	8	11

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125MT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.032 OD x 9.000 L x 0.208 in thick; Cylinder Weight 1461 grams

Test terminated without failure after 10 pressure cycles to 10,000 psi

Maximum compressive hoop stress at test termination: 145,000 psi

Table 7-1 Strains on Dow Ceramic Cylinder PN 55910-0126847 SN# 7
under Short Term Pressurizations

Pressure (Psi)	Interior Gage Locations Location A - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-330	-117	-306	-84	-290	-124	-314	-97	-283	-104
2000	-641	-221	-600	-184	-585	-234	-626	-194	-570	-210
3000	-938	-323	-886	-277	-875	-341	-917	-293	-853	-309
4000	-1260	-431	-1195	-381	-1189	-454	-1226	-398	-1170	-416
5000	-1557	-533	-1478	-477	-1488	-557	-1511	-497	-1449	-512
6000	-1872	-639	-1776	-580	-1801	-664	-1812	-600	-1748	-618
7000	-2176	-746	-2064	-677	-2110	-769	-2101	-702	-2037	-716
8000	-2491	-847	-2351	-775	-2416	-875	-2390	-803	-2329	-818
9000	-2785	-945	-2621	-867	-2706	-974	-2664	-899	-2601	-915
10000	-3114	-1051	-2909	-970	-3025	-1081	-2965	-1004	-2893	-1022
11000	-3435	-1151	-3193	-1072	-3328	-1187	-3269	-1105	-3166	-1126
12000	-3785	-1256	-3482	-1178	-3650	-1294	-3582	-1207	-3432	-1235
13000	-4092	-1346	-3726	-1267	-3938	-1391	-3859	-1298	-3669	-1331
14000	-4444	-1445	-4001	-1373	-4261	-1499	-4163	-1398	-3934	-1438
15000	-4803	-1540	-4262	-1475	-4592	-1599	-4459	-1496	-4185	-1546
16000	-5164	-1632	-4504	-1578	-4921	-1697	-4739	-1592	-4419	-1651
17000	-5531	-1715	-4715	-1675	-5255	-1787	-5003	-1684	-4624	-1750
18000	-5969	-1800	-4918	-1791	-5639	-1884	-5295	-1782	-4813	-1866
18500	-6221	-1841	-4997	-1852	-5854	-1929	-5441	-1833	-4885	-1925
19000	-6456	-1868	-5043	-1905	-6047	-1967	-5566	-1872	-4926	-1980
19500	-6767	-1900	-5071	-1973	-6302	-2010	-5716	-1923	-4947	-2047
19800									-4949	
0	2	0	-23	-2	8	-3	-26	0	-6	0
10000	-3190	-1027	-2846	-942	-3046	-1074	-2972	-964	-2785	-1014
0	1	0	-10	-1	3	-1	-4	1	-1	0

NOTES: All strains are in microinches per inch
 Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09
 Ceramic Composition: Boron Carbide Aluminum Composite
 End Closures: Plane Steel Bulkheads providing radial support
 Cylinder Dimensions: 6.032 OD x 9.000 L x 0.208 in thick; Cylinder Weight 1461 grams
 Test terminated without failure after pressurization to 19,800 psi
 Maximum compressive hoop stress at test termination: 287,100 psi

Test Date: 4-25-92

ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-0126847 SN# 07

Pressurizations 1-10

Pressure	Events	Time	Notes:
0000	0	9:13	1. Transducer: AET AC175
1000	2-2	9:15	SN# 7799 5 to 200 KHZ
2000	6-6	9:16	2. Amplifier Setting:
3000	10-10	9:18	Rate: T
4000	10-10	9:20	Gain: 60 DB
5000	16-16	9:22	Threshold: Automatic
6000	16-16	9:25	Function: Events
7000	17-17	9:26	3: Recorder:
8000	19-19	9:30	Channel "A" Events,
9000	20-21	9:32	4000 Full Range
10000	22-22	9:34	Channel "B" Rms,
TENTH PRESSURE CYCLE			50 MV Full Scale,

0000	0	10:00	0.5 CM/Min Chart Scale
1000	0-0	10:01	
2000	0-0	10:02	
3000	0-0	10:03	
4000	0-0	10:04	
5000	1-1	10:05	
6000	2-2	10:06	
7000	2-2	10:07	
8000	2-2	10:08	
9000	2-2	10:09	
10000	2-2	10:10	

Test terminated after 10 cycles to 10,000 psi without failure

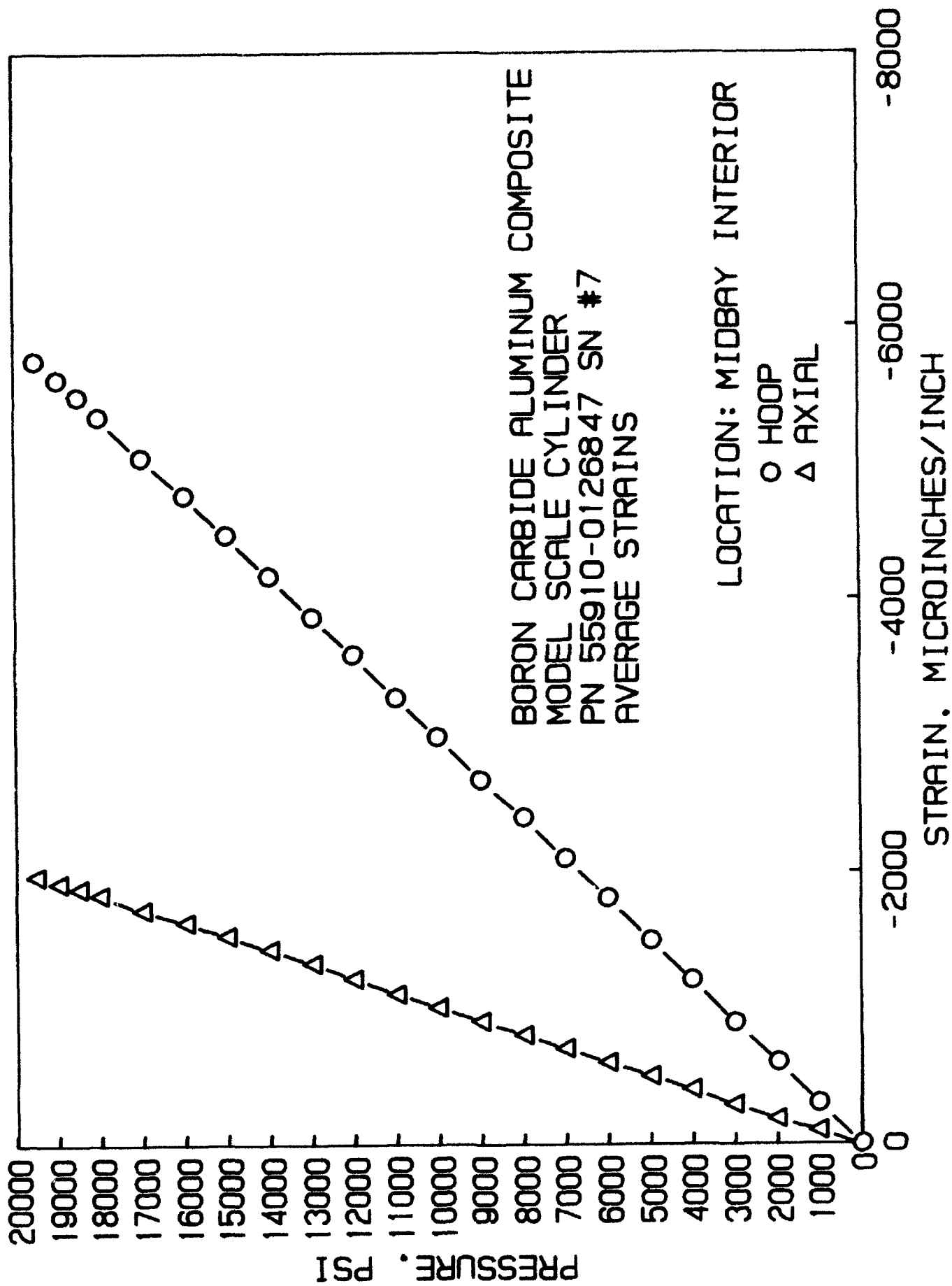
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER 55910-0126847 SN# 07

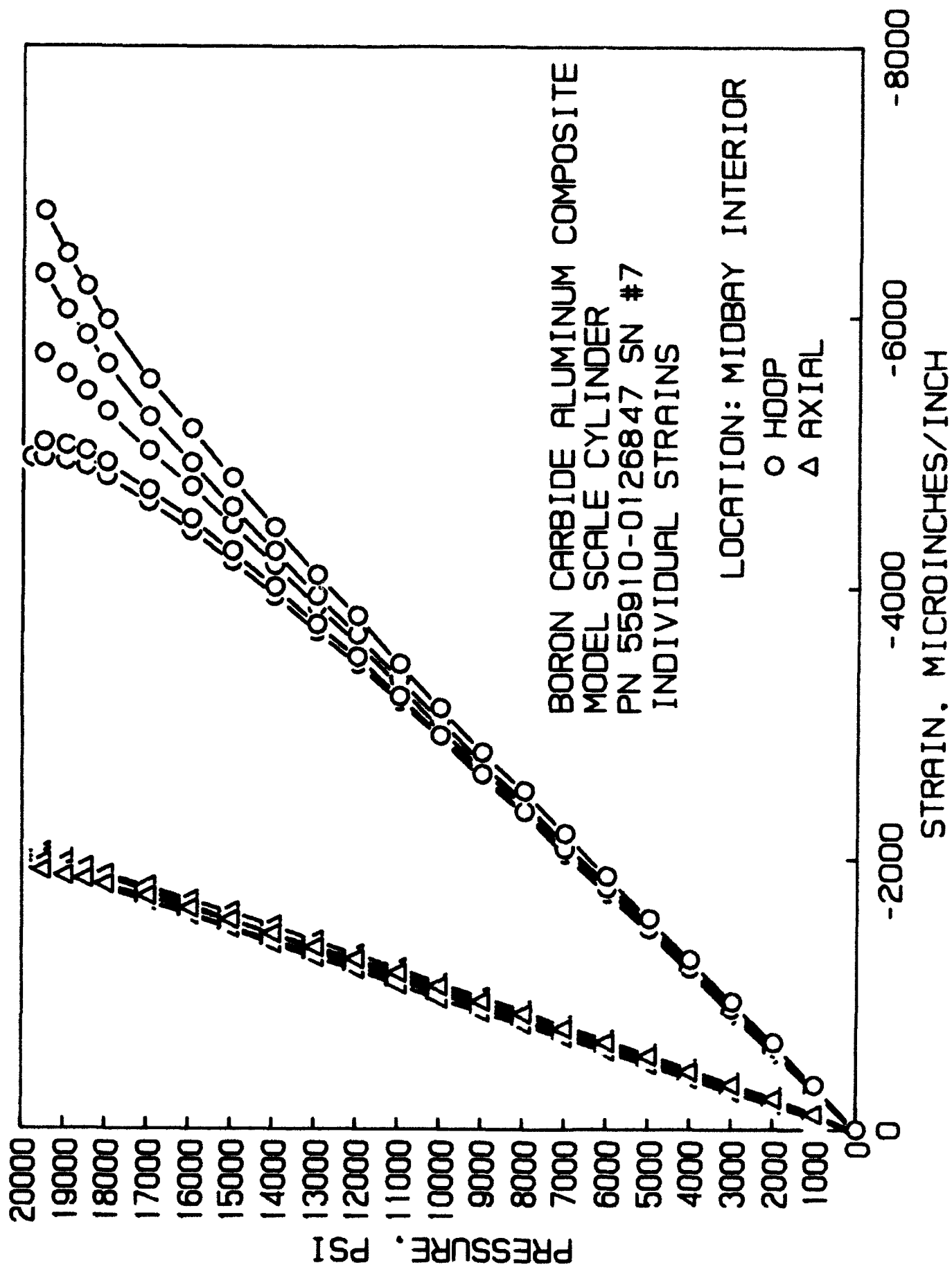
Pressurizations 1 and 2

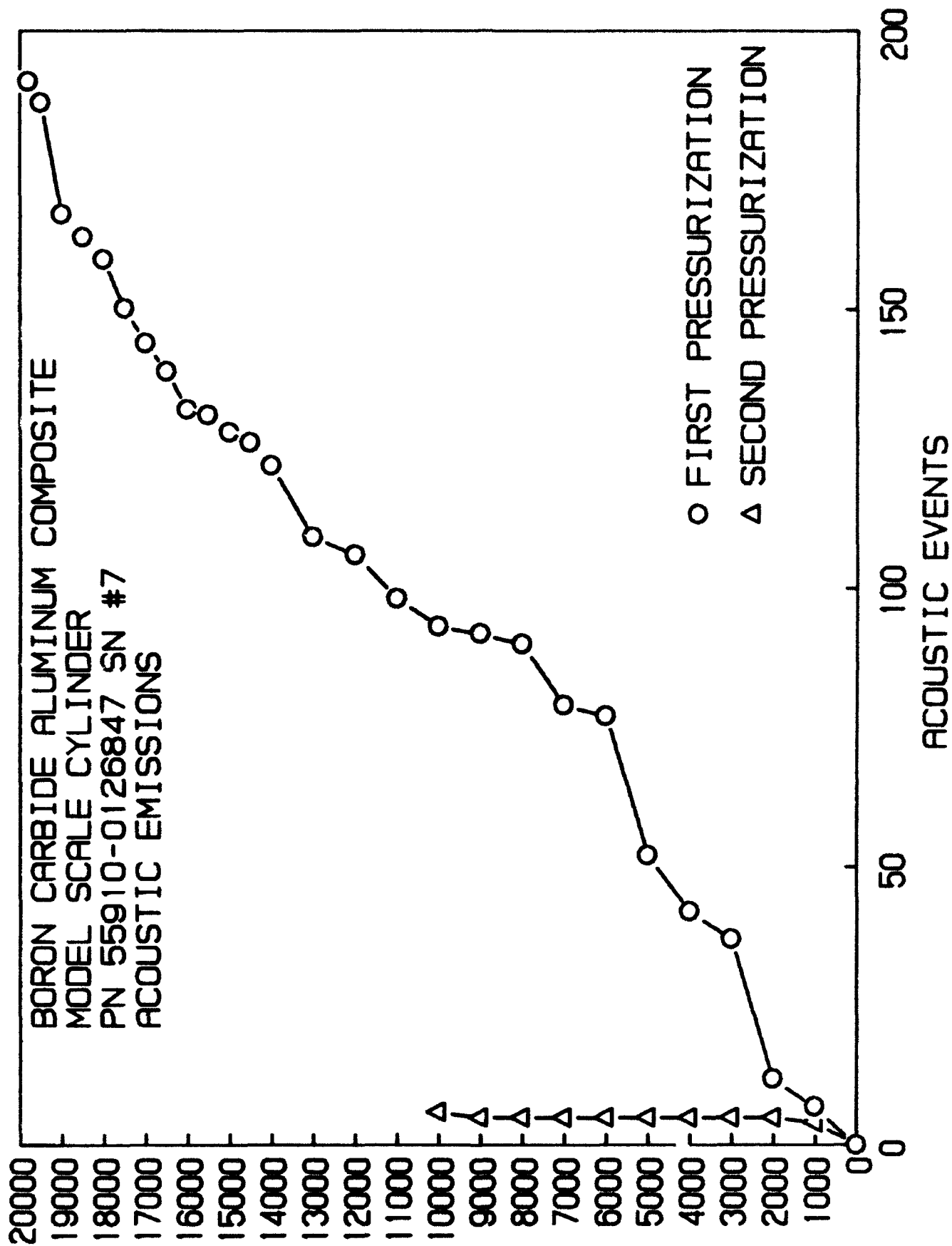
Pressure	Events	
	Pressurization 1	2
0000	0	0
1000	7	4
2000	12	5
3000	37	5
4000	42	5
5000	52	5
6000	77	5
7000	79	5
8000	90	5
9000	92	5
10000	93	6
11000	98	
12000	106	
13000	109	
14000	122	
14500	126	
15000	128	
15500	131	
16000	132	
16500	139	
17000	144	
17500	150	
18000	159	
18500	163	
19000	167	
19500	187	
19800	191	

Test terminated at 19,800 psi without implosion

- Notes:
1. Transducer: AET AC175 SN#7799
5 to 200 KHZ
 2. Amplifier Setting:
Rate: T Gain: 60 DB
Threshold: Automatic
Function: Events
 3. Recorder:
Channel "A" Events,
400 Full Range,
Channel "B" Rms,
50 MV Full Scale,
0.5 CM/Min Chart Speed







Test Cylinder SN#8 Type 3

Table 8 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 8
under Short Term Pressurizations

Interior Gage Locations

Location A - Midbay

Pressure (Psi)	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-454	-170	-419	-126	-475	-151	-444	-108	-454	-162
2000	-926	-333	888	-286	-946	-308	-920	-271	-935	-324
3000	-1393	-483	-1328	-440	-1374	-457	-1390	-433	-1365	-454
4000	-1873	-634	-1802	-600	-1833	-615	-1888	-592	-1832	-612
5000	-2301	-768	-2261	-755	-2265	-759	-2342	-743	-2331	-769
6000	-2776	-927	-2744	-920	-2739	-924	-2837	-906	-2821	-930
7000	-3242	-1091	-3192	-1072	-3185	-1078	-3340	-1067	-3253	-1123
8000	-3856	-1315	-3604	-1234	-3711	-1227	-4038	-1265	-3528	-1366
9000	-4324	-1442	-4063	-1383	-4192	-1377	-4498	-139	-4013	-1553
10000	-4799	-1578	-4564	-1551	-4695	-1541	-5006	-1542	-4505	-1737
11000	-5253	-1718	-5148	-1723	-5207	-1715	-5511	-1701	-5112	-1904
12000	-5602	-1893	-5699	-1889	-5719	-1888	-6028	-1866	-5817	-2019
12500	-5804	-1999	-5874	-1991	-5998	-1992	-6359	-1963	-6229	-2084
13000	-5921	-2094	-6020	-2066	-6224	-2067	-6677	-2033	-6573	-2133
13200	-5853	-2142	-5948	-2119	-6356	-2114	-7049	-2058	-6713	-2122
13400	-5794	-2189	-5943	-2152	-6465	-2145	-7241	-2081	-6363	-2115
0	-80	-43	-99	-31	-28	16	-158	-46	-21	-24

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Titanium Hemispherical Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 4.805 L x 0.137 in thick

Cylinder Weight: 577 grams

Test terminated without failure at 13,500 psi

Maximum compressive hoop stress at test termination: 295,000 psi

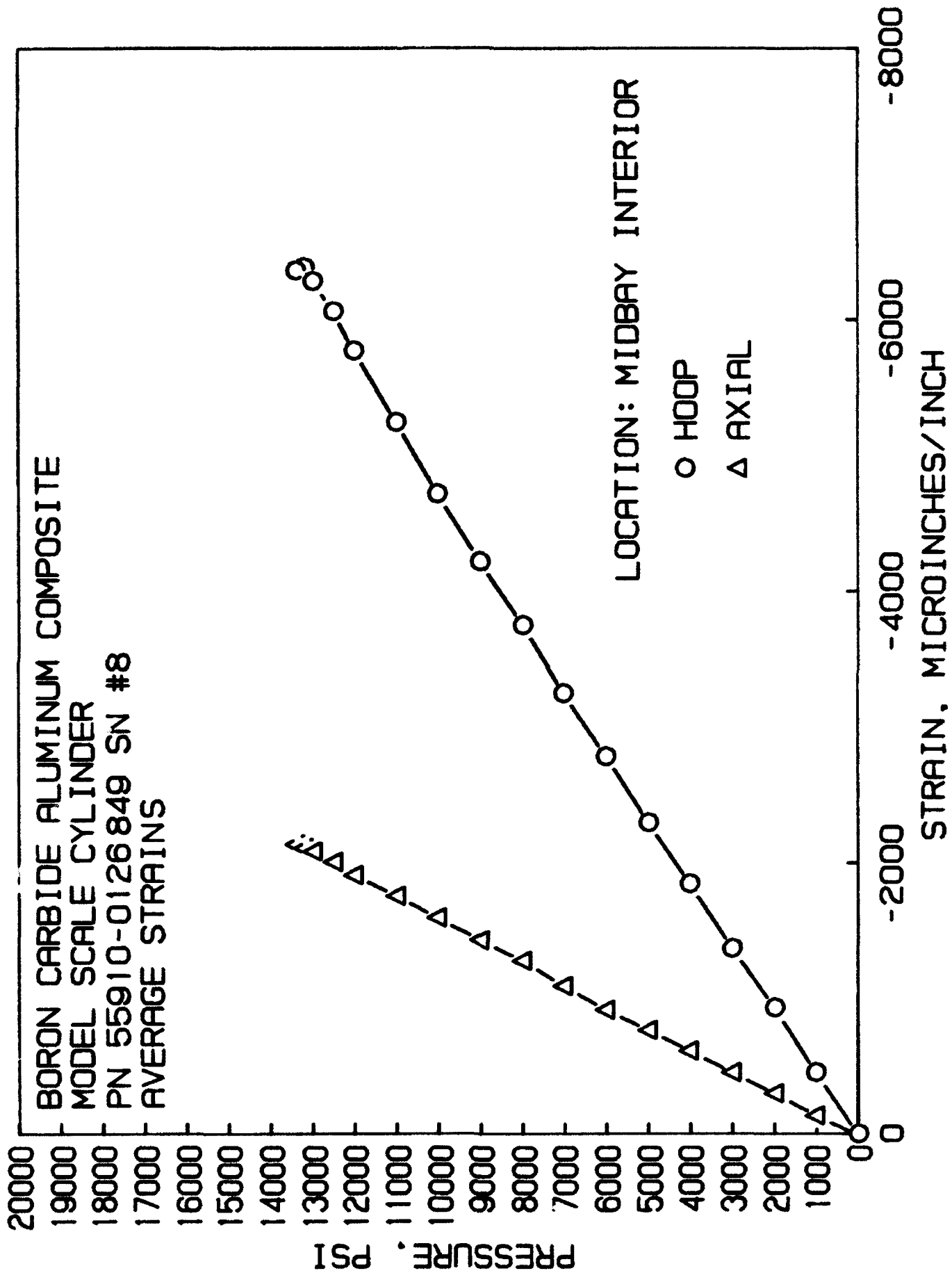
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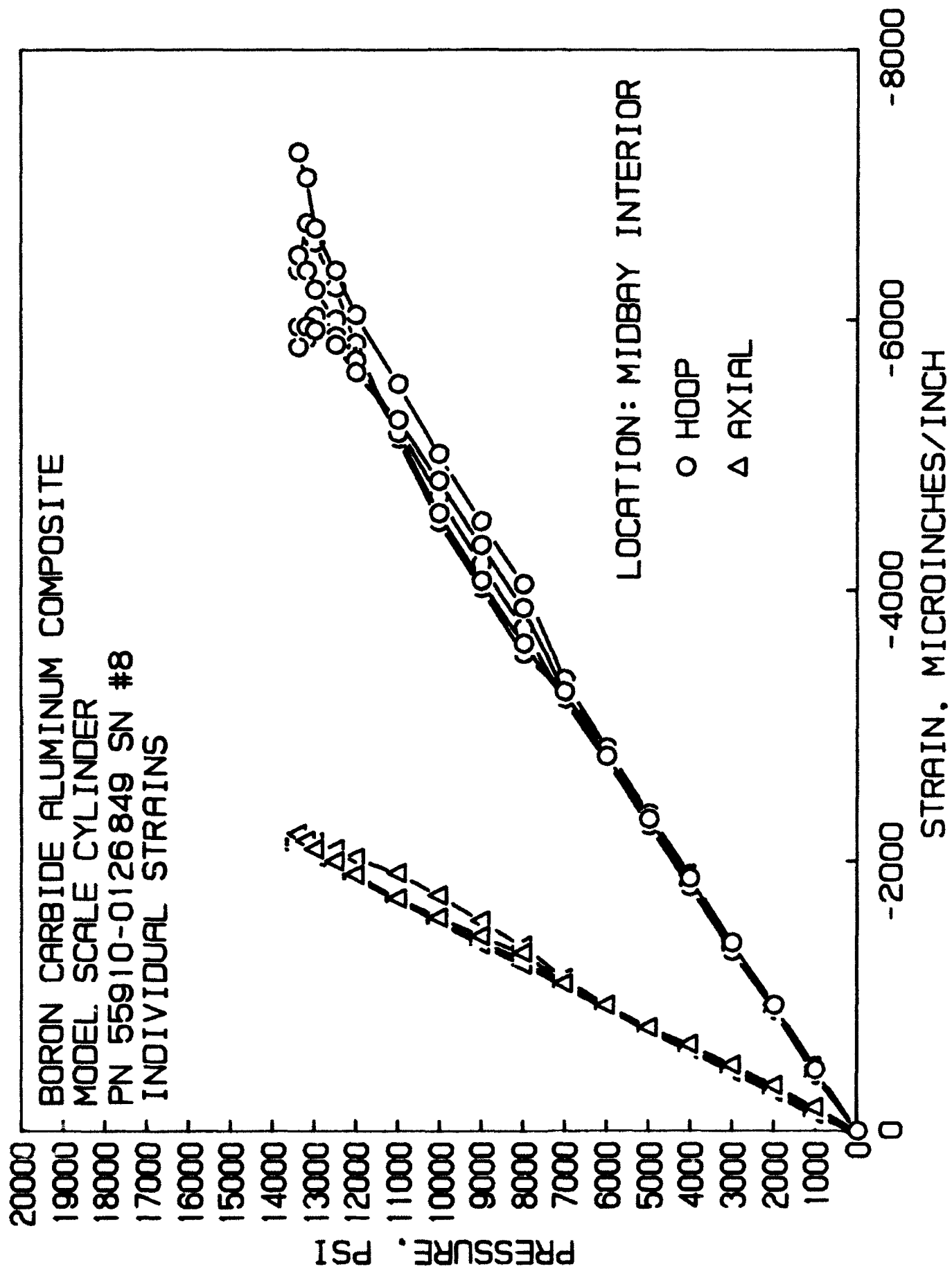
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-0126849 SN# 08

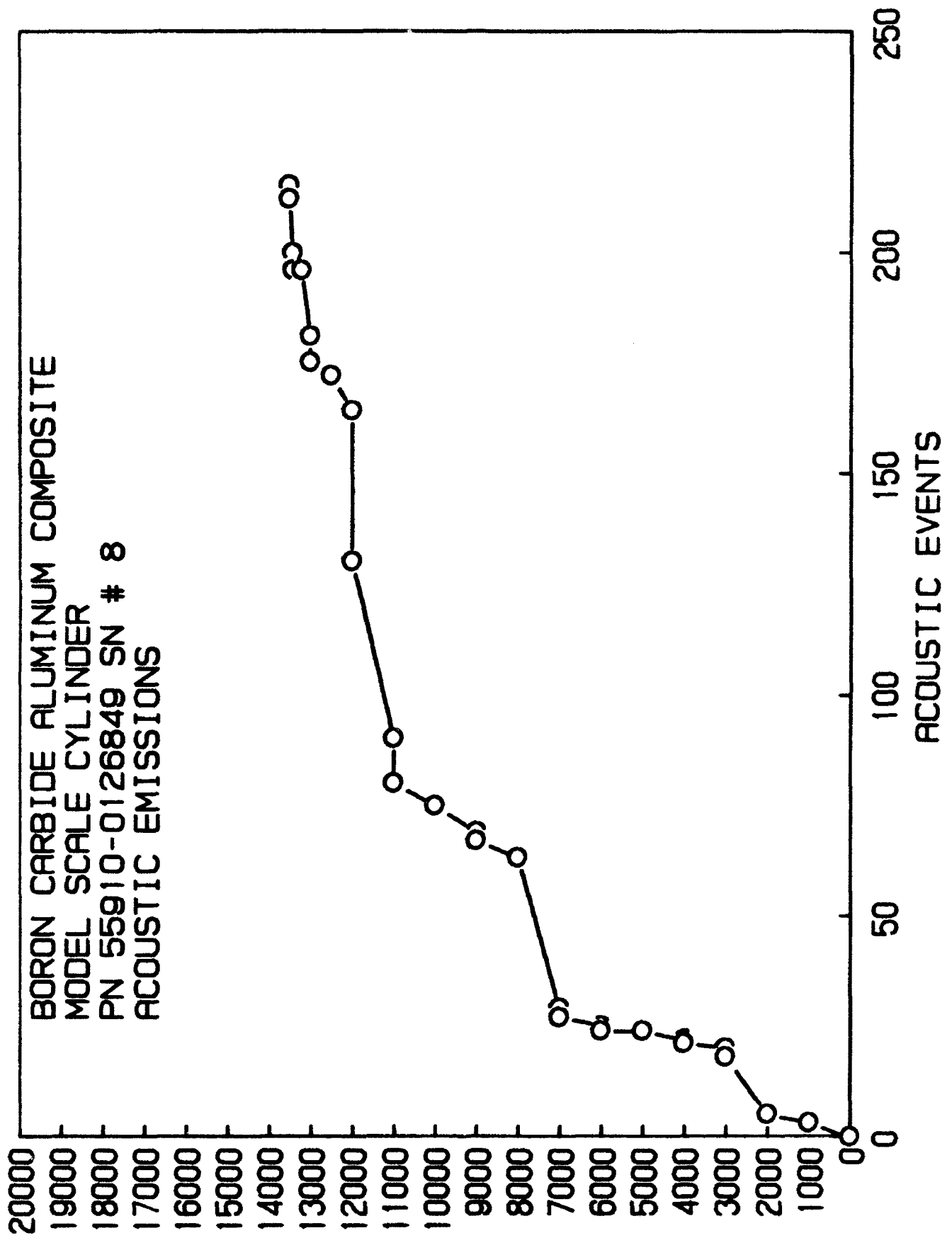
Pressurization # 1

Pressure	Events	Time	Notes:
0000	0	5:10	1. Transducer: AET AC175
1000	3-3	5:13	SN# 7799 5 to 200 KHZ
2000	5-5	5:15	2. Amplifier Setting:
3000	18-20	5:16	Rate: T
4000	21-22	5:18	Gain: 60 DB
5000	24-24	5:20	Threshold: Automatic
6000	24-25	5:21	Function: Events
7000	27-29	5:23	3. Recorder:
8000	63-63	5:25	Channel "A" Events,
9000	67-69	5:26	4000 Full Range
10000	75-75	5:30	Channel "B" Rms,
11000	80-90	5:32	50 MV Full Scale,
12000	130-164	5:33	0.5 CM/Min Chart Scale
12500	172-172	5:35	
13000	175-181	5:37	
13200	196-196	5:40	
13400	196-200	5:41	
13500	212-215	5:43	

Failed at 13,500 psig







Test Cylinder SN#9 Type 3

Table 9 Strains on Dow Ceramic Cylinder PN 55910-0126849 SN# 9
under Short Term Pressurizations

Interior Gage Locations

Pressure (Psi)	Location A - Midbay									
	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-377	-295	-583	-10	-447	-159	-507	-181	-485	-47
2000	-849	-477	-1003	-150	-847	-349	-995	-332	-980	-162
3000	-1322	-631	-1438	-301	-1279	-519	-1467	-488	-1454	-308
4000	-1768	-787	-1871	-453	-1693	-679	-1910	-641	-1905	-473
5000	-2221	-949	-2332	-610	-2143	-854	-2383	-807	-2363	-625
6000	-2651	-1101	-2769	-755	-2568	-1003	-2826	-947	-2796	-770
7000	-3075	-1241	-3206	-889	-2990	-1154	-3270	-1096	-3232	-902
8000	-3527	-1403	-3685	-1046	-3449	-1321	-3735	-1253	-3691	-1066
9000	-3964	-1562	-4129	-1197	-3893	-1480	-4176	-1403	-4118	-1215
10000	-4369	-1711	-4569	-1345	-4342	-1639	-4636	-1570	-4572	-1380
11000	-4834	-1883	-5045	-1512	-4822	-1803	-5107	-1731	-5020	-1538
12000	-5254	-2032	-5495	-1664	-5284	-1961	-5555	-1886	-5449	-1690
13000	-5686	-2191	-5976	-1827	-5777	-2126	-6027	-2046	-5890	-1849
13500	-5912	-2269	-6232	-1910	-6037	-2210	-6273	-2128	-6119	-1933
14000	-6113	-2336	-6463	-1983	-6276	-2288	-6499	-2203	-6326	-2005
14500	-6353	-2416	-6733	-2072	-6551	-2377	-6762	-2288	-6567	-2091
15000	-6571	-2484	-6970	-2149	-6792	-2457	-7003	-2365	-6791	-2168
15500	-6795	-2564	-7215	-2237	-7039	-2544	-7251	-2448	-7015	-2250
16000	-7020	-2637	-7467	-2319	-7297	-2660	-7700			

NOTES: ALL strains are in microinches per inch

Electric resistance strain gages are CEA-06-125UT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Plane Steel Bulkheads providing radial support

Cylinder Dimensions: 6.000 OD x 4.81 L x 0.139 in thick

Cylinder Weight: 572 grams

Catastrophic failure at 16,000 psi

Maximum compressive hoop stress at test termination: 345,000 psi

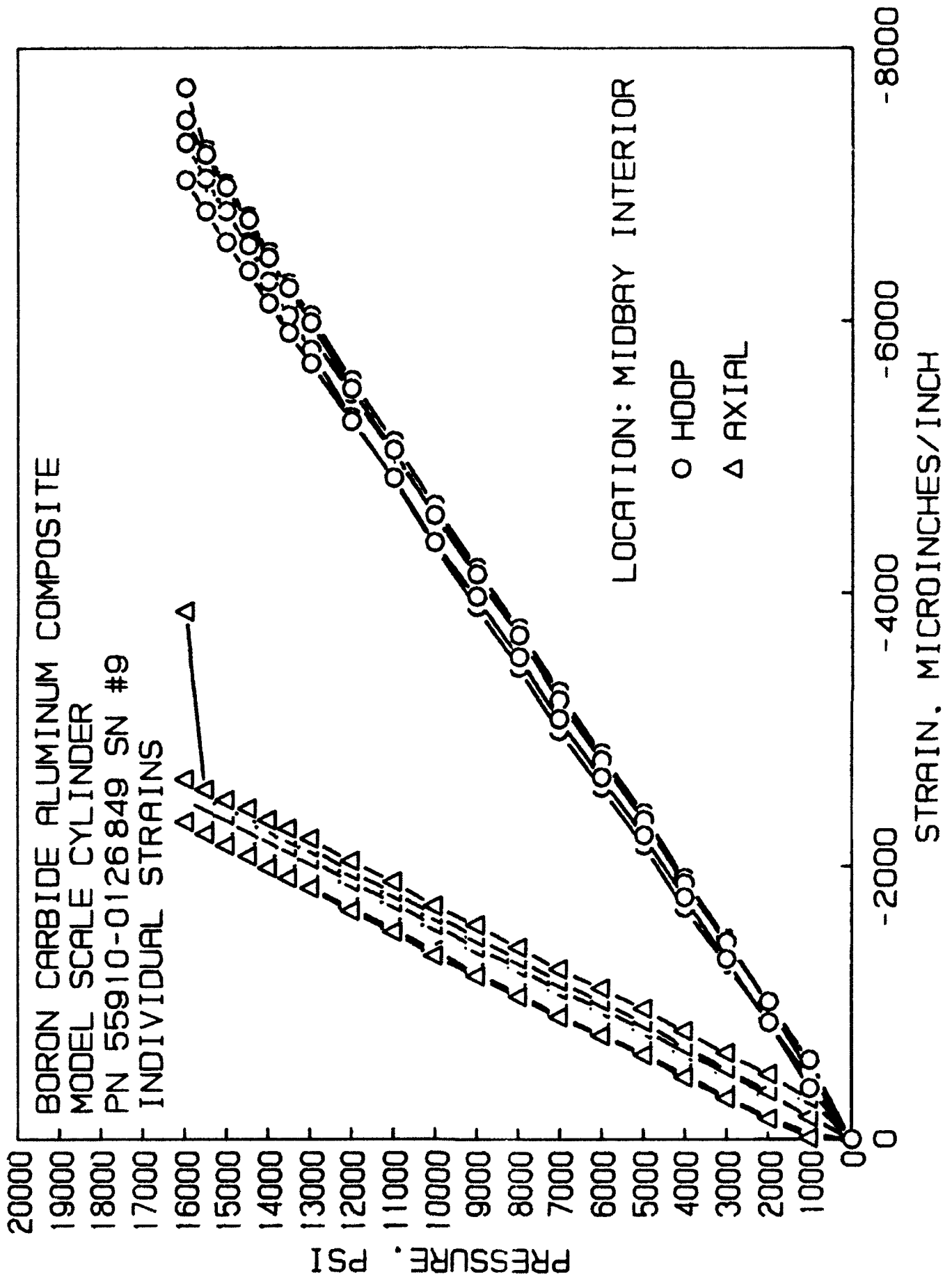
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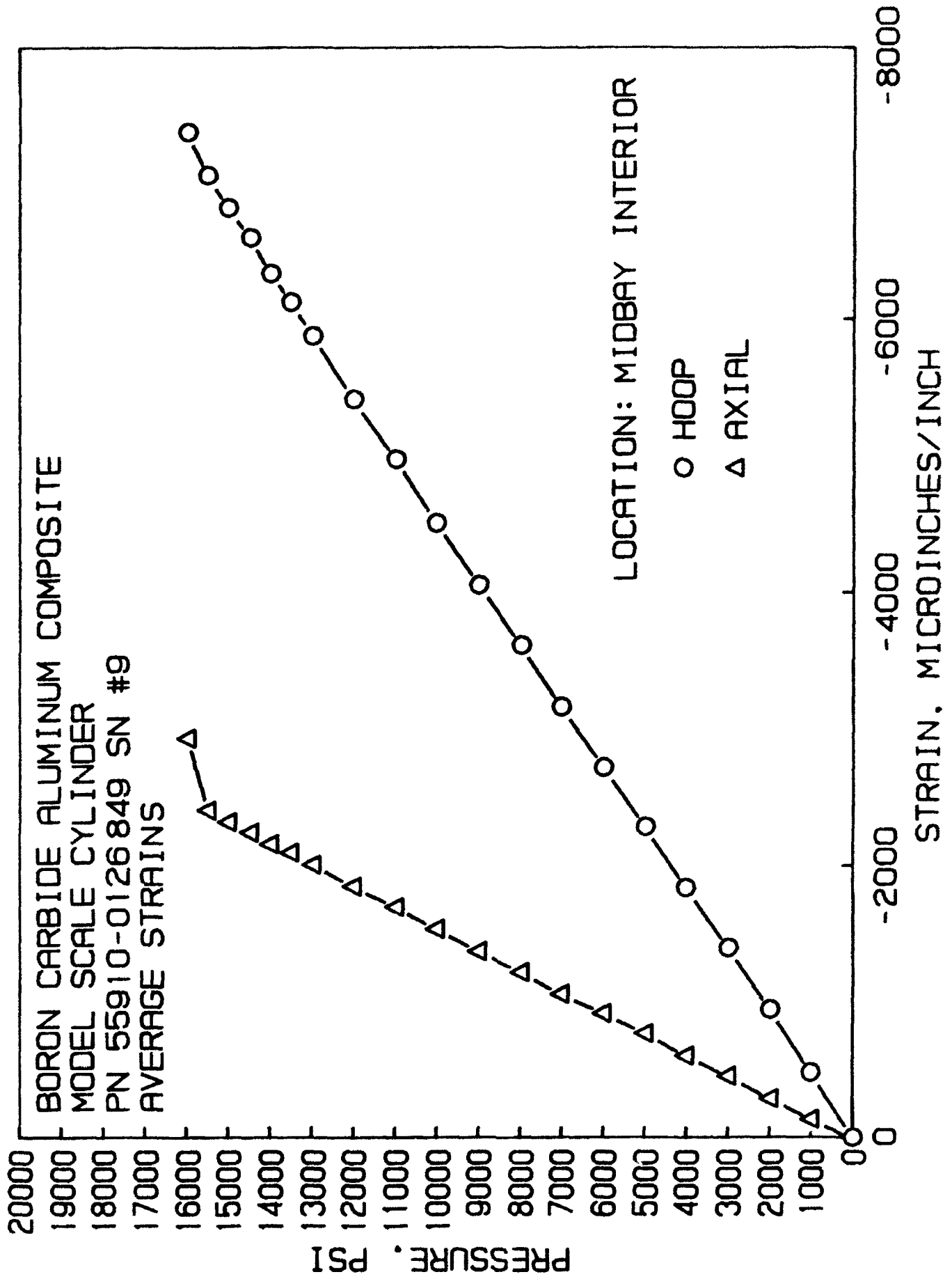
ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER DOW PN 55910-0126849 SN# 09

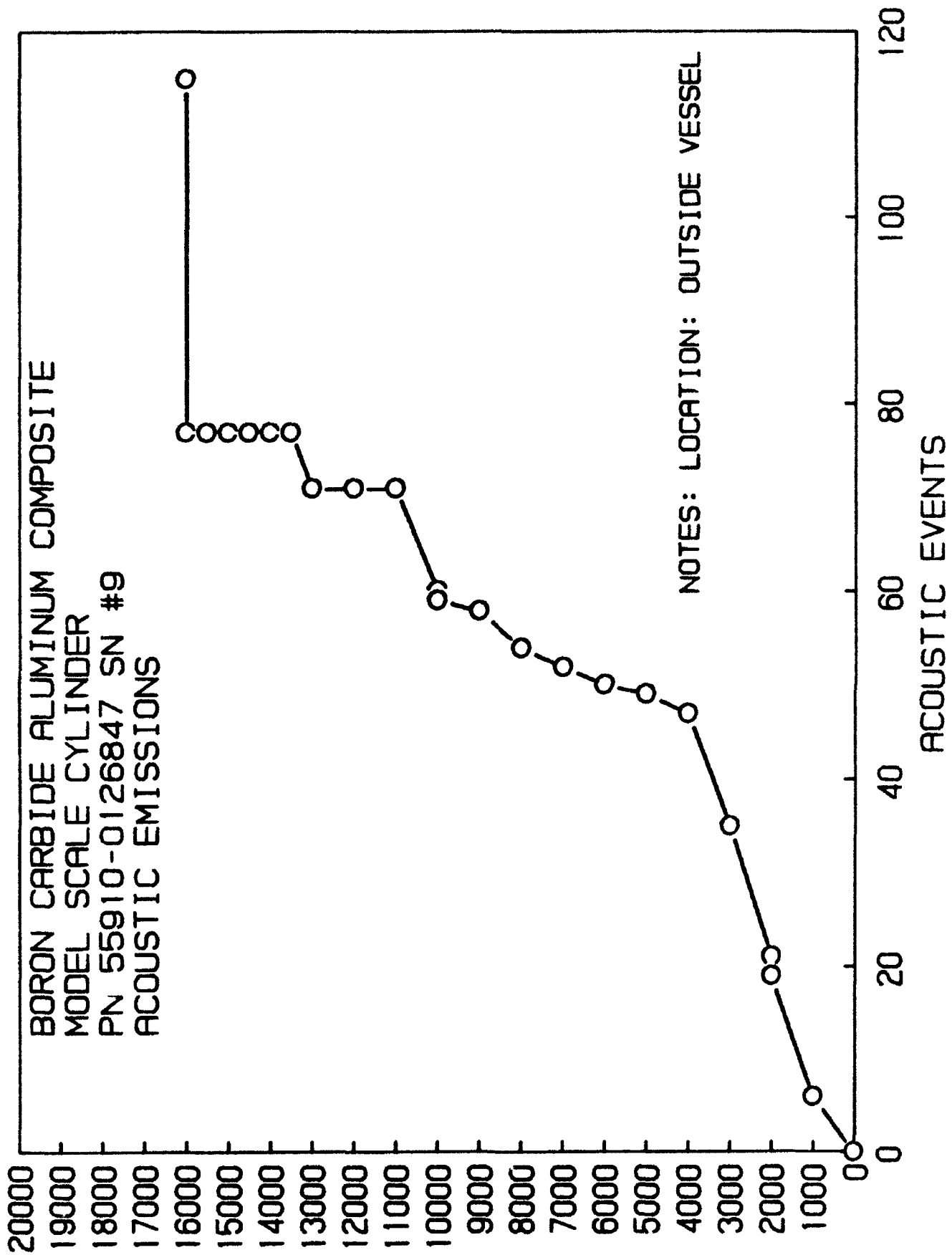
Pressurization # 1

Pressure	Events	Time	Notes:
0000	0	12:38	1. Transducer: AET AC175L
1000	6-6	12:42	SN# 7808 5 to 200 KHZ
2000	19-21	12:44	2. Amplifier Setting:
3000	35-35	12:46	Rate: T
4000	47-47	12:47	Gain: 60 DB
5000	49-49	12:49	Threshold: Automatic
6000	50-50	12:52	Function: Events
7000	52-52	12:55	Scale: 1
8000	54-54	12:56	3. Recorder:
9000	58-58	12:59	Channel "A" Events
10000	59-60	1:00	Full Range 4000
11000	71-71	1:03	Channel "B" Rms
12000	71-71	1:05	50 MV Full Scale
13000	71-71	1:07	0.5 CM/ Min Chart Speed
13500	77-77	1:09	
14000	77-77	1:10	
14500	77-77	1:12	
15000	77-77	1:15	
15500	77-77	1:16	
16000	77-115	1:20	

Failed at 16,000 psig







Test Cylinder SN#10 Type 1

Table 10. Tested with plane steel bulkheads

Table 10.1. Tested with titanium hemispherical bulkheads

Table 10 Strains on Dow Ceramic Cylinder PN 55910-0126847 SN# 10
under Short Term Pressurizations

Interior Gage Locations

Location A - Midbay

Pressure (Psi)	1		2		3		4		5	
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
1000	-269	-140	-320	-106	-332	-82	-260	-153	-396	-45
2000	-579	-259	-607	-193	-649	-198	-543	-258	-712	-138
3000	-897	-377	-902	-291	-977	-307	-838	-367	-1019	-240
4000	-1201	-489	-1186	-393	-1298	-409	-1116	-474	-1327	-338
5000	-1513	-606	-1490	-503	-1640	-516	-1395	-585	-1672	-440
6000	-1809	-718	-1793	-606	-1969	-622	-1670	-695	-1994	-542
7000	-2104	-831	-2106	-711	-2295	-727	-1960	-809	-2322	-644
8000	-2396	-943	-2418	-817	-2606	-834	-2260	-917	-2641	-745
9000	-2699	-1053	-2736	-922	-2917	-942	-2558	-1030	-2953	-851
10000	-2987	-1162	-3041	-1024	-3225	-1044	-2850	-1135	-3257	-952
11000	-3297	-1277	-3358	-1135	-3546	-1154	-3160	-1242	-3575	-1057
12000	-3582	-1379	-3650	-1234	-3846	-1255	-3439	-1346	-3870	-1157
13000	-3883	-1489	-3956	-1340	-4168	-1362	-3731	-1454	-4182	-1262
14000	-4163	-1593	-4247	-1438	-4471	-1463	-4003	-1555	-4478	-1358
15000	-4449	-1705	-4550	-1542	-4786	-1572	-4282	-1660	-4788	-1461
16000	-4718	-1810	-4851	-1636	-5088	-1675	-4541	-1761	-5094	-1559
17000	-4989	-1923	-5172	-1739	-5408	-1786	-4801	-1868	-5435	-1657
18000	-5214	-2034	-5492	-1830	-5721	-1888	-5017	-1971	-5784	-1745
18500	-5318	-2097	-5680	-1881	-5905	-1943	-5124	-2032	-6001	-1793
19000	-5400	-2154	-5849	-1925	-6077	-1922	-5197	-2089	-6210	-1833
19500	-5446	-2215	-6030	-1965	-6266	-2034	-5238	-2148	-6459	-1865
0	18	5	-20	19	19	7	-13	21	16	17
10000	-2895	-1173	-3148	-988	-3115	-1082	-2876	-1108	-3270	-963
18500	-5223	-2109	-5750	-1878	-5878	-1956	-5106	-2031	-6059	-1807
19000	-5332	-2167	-5922	-1928	-6068	-2006	-5201	-2095	-6275	-1852
19500	-5388	-2215	-6054	-1966	-6221	-2041	-5248	-2144	-6461	-1882
10000	-2875	-1181	-3182	-995	-2132	-1091	-2877	-1110	-3302	-970
0	-2	-4	-4	-6	-3	-7	-5	-5	5	-11

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

End Closures: Plane Steel Bulkheads providing radial support

Cylinder Dimensions: 6.039 OD x 9.00 L x 0.206 in thick

Cylinder Weight: 1451 grams

Test terminated at 19,500 psi without implosion

Maximum compressive hoop stress at test termination: 285,000 psi

Test Date: 7-12-92

ACOUSTIC EMISSIONS DURING HYDROSTATIC TESTING
OF MODEL CYLINDER 55910-0126847 SN# 10

Pressurizations 1 and 2

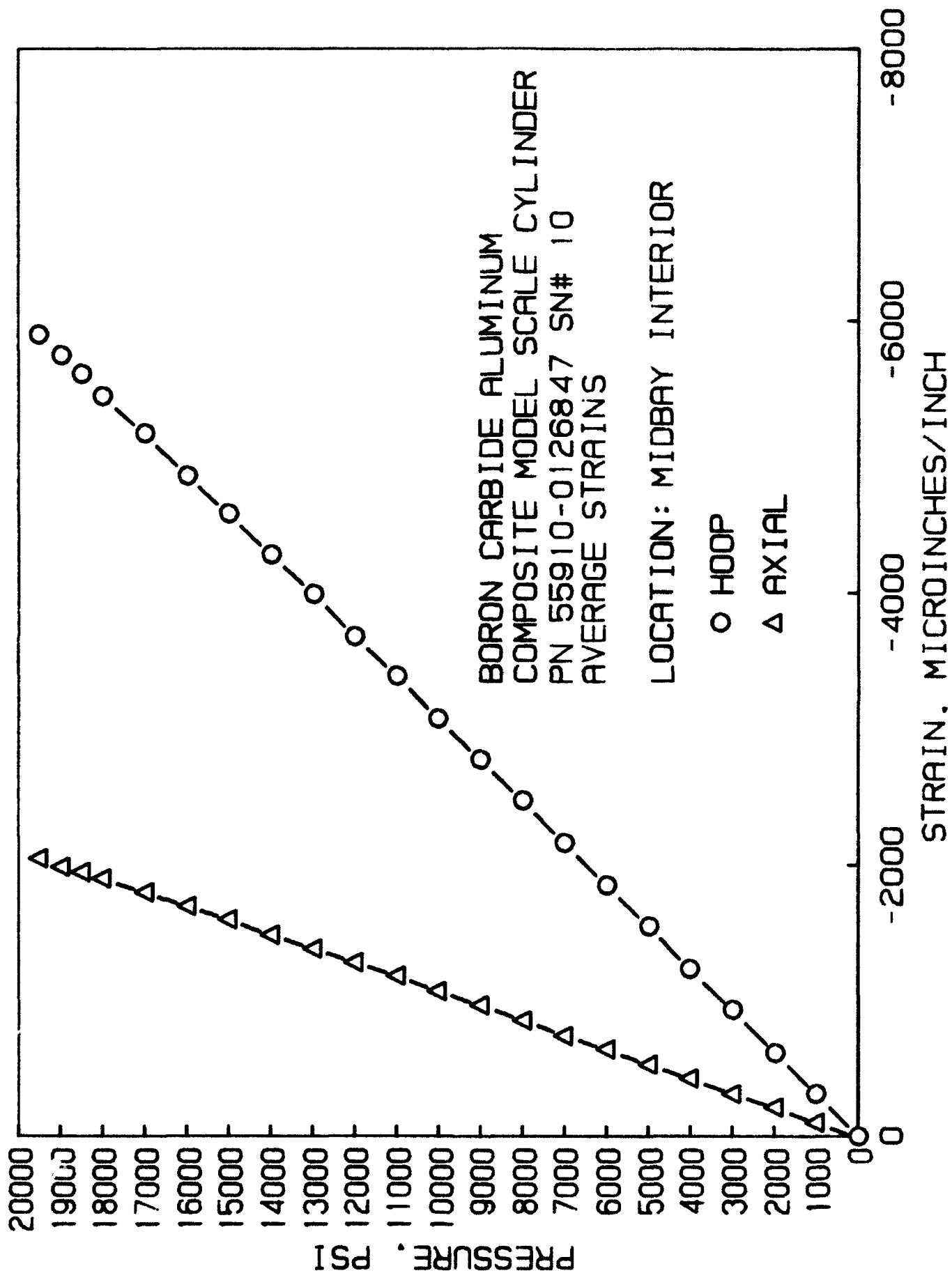
Pressure	Events	
	Pressurization 1	2
0000	0	0
1000	9	0
2000	17	0
3000	20	0
4000	22	0
5000	50	0
6000	50	0
7000	57	0
8000	61	0
9000	67	0
10000	73	5
11000	81	15
12000	85	15
13000	86	15
14000	86	15
15000	89	15
16000	90	17
17000	112	35
18000	112	38
18500	117	38
19000	118	38
19500	118	38

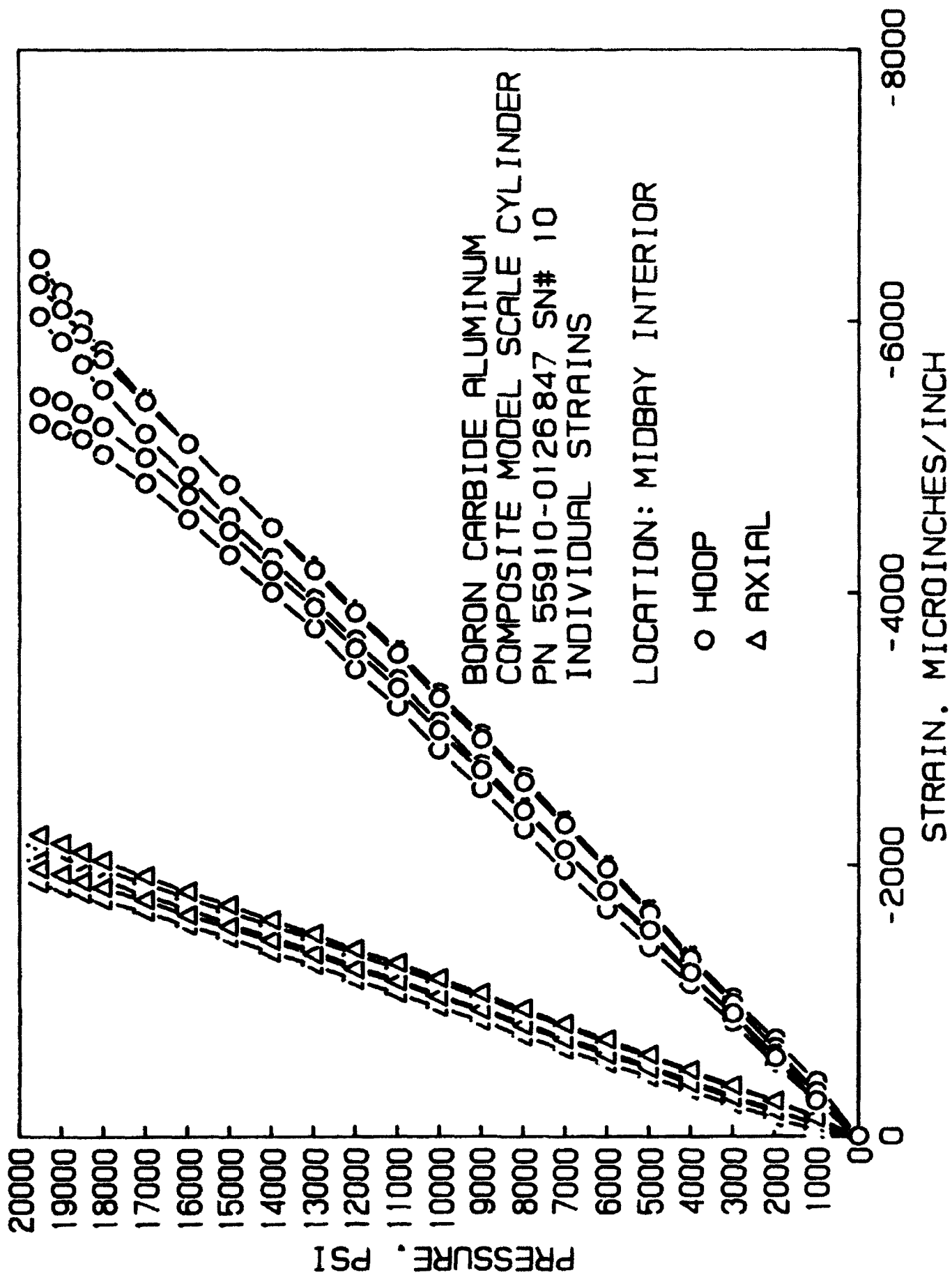
Test terminated without failure at 19,500 psig.

Notes: 1. Transducer: AET AC175 SN#8686
5 to 200 KHZ

2. Amplifier Setting:
Rate: T Gain: 60 DB
Threshold: Automatic
Function: Events
Scale: 1

3. Recorder:
Channel "A" Events,
400 Full Range,
Channel "B" Rms,
50 MV Full Scale,
0.5 CM/Min Chart Speed





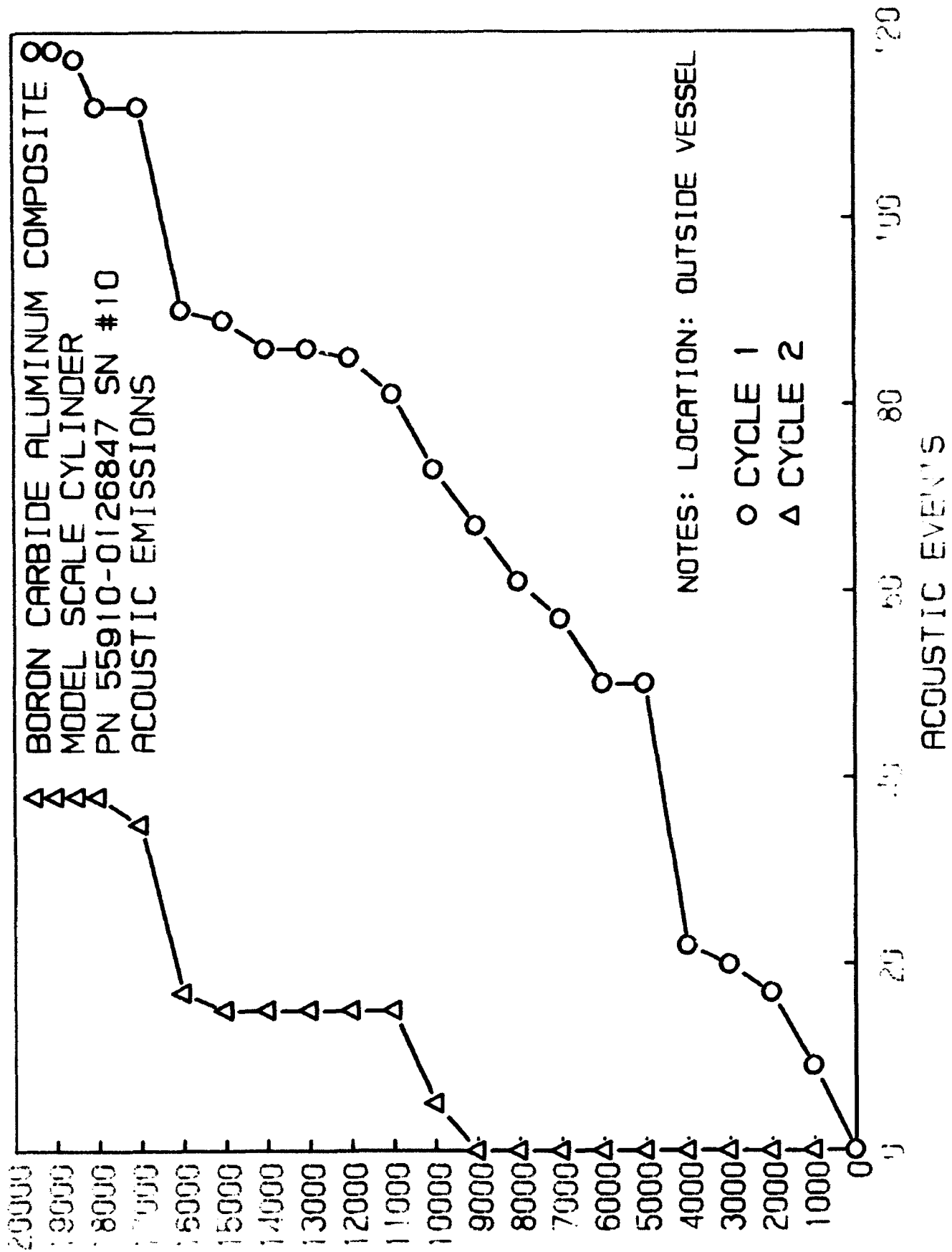


Table 10-1 Strains on Dow Ceramic Cylinder PN 55910-0126847 SN# 10
under Short Term Pressurization

Pressure (Psi)	Interior Gage Locations									
	Location A - Midbay									
	1	2	3	4	5					
	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial	Hoop	Axial
0	0	0	0	0	0	0	0	0	0	0
2000	-820	-338	-272	-798	-255	-236	-592	-224	-592	-224
4000	-1220	-413	-431	-1217	-429	-428	-1225	-432	-1225	-432
6000	-1940	-644	-768	-1886	-659	-653	-1897	-672	-1897	-672
8000	-2456	-879	-915	-2473	-869	-869	-2435	-908	-2435	-908
10000	-3060	-1094	-1108	-3108	-1110	-1088	-3146	-1108	-3146	-1108
12000	-4141	-1320	-1318	-4181	-1336	-1340	-3708	-1334	-3708	-1334
14000	-4207	-1529	-1538	-4854	-1593	-1573	-4338	-1514	-4338	-1514
15000	-4773	-2138	-2130	-4953	-2148	-2113	-5160	-2085	-5160	-2085
15700	-3921									

NOTES: All strains are in microinches per inch

Electric resistance strain gages are CEA-06-125WT-350, Gage Factor 2.09

Ceramic Composition: Boron Carbide Aluminum Composite

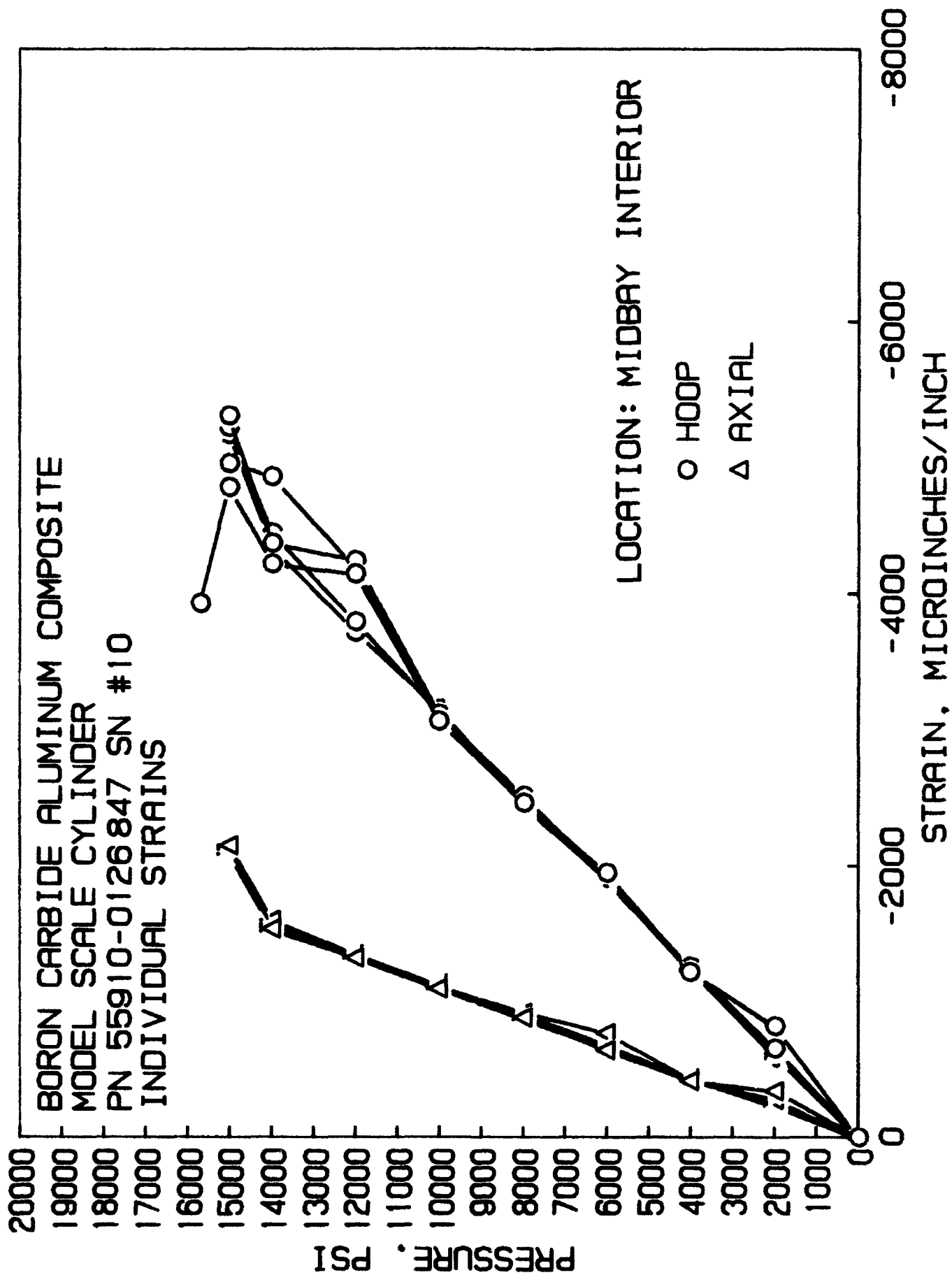
End Closures: Titanium Hemispherical Bulkheads providing radial support

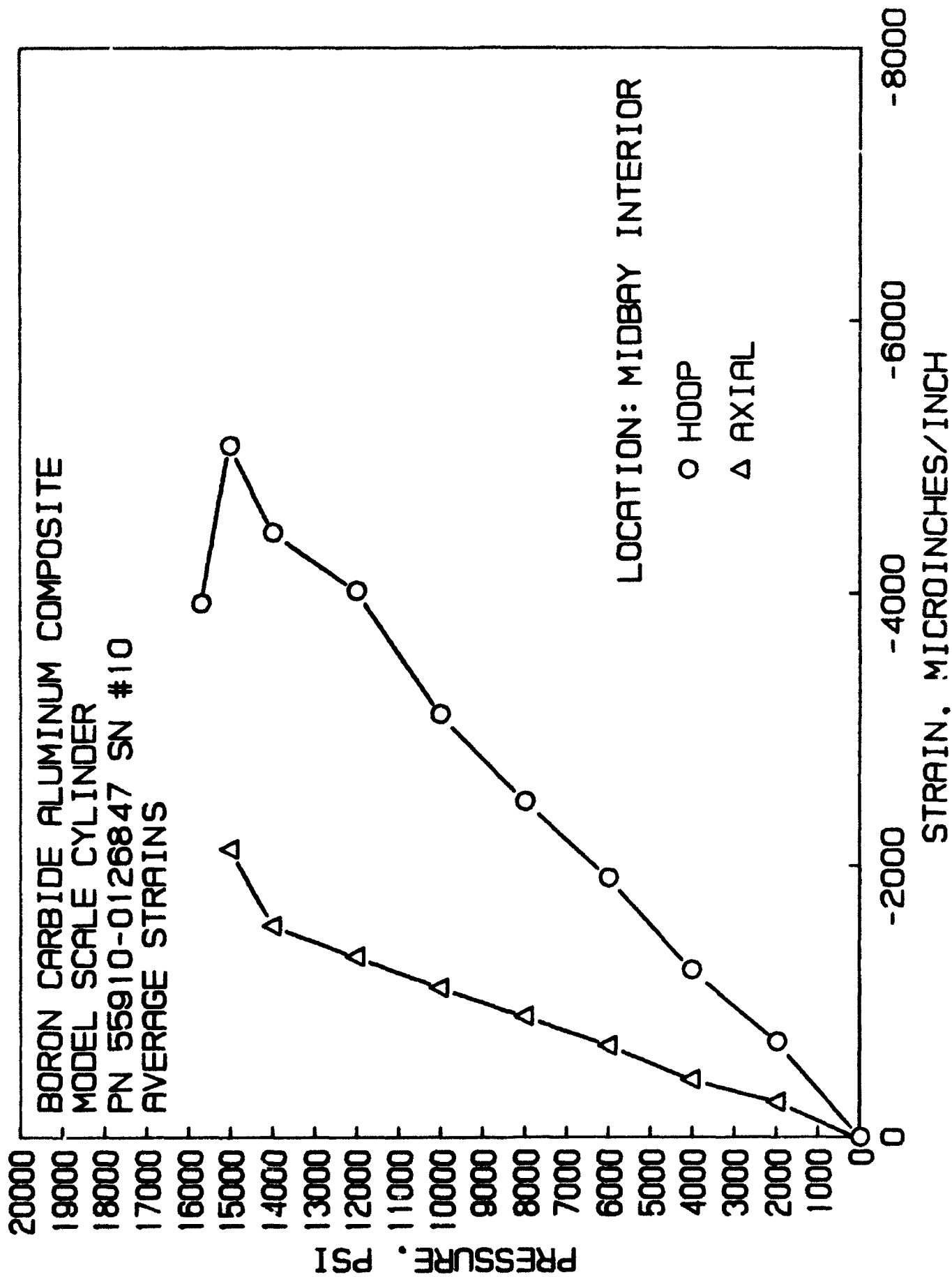
Cylinder Dimensions: 6.039 OD x 9.00 L x 0.206 in thick

Cylinder Weight: 1451 grams

Catastrophic Implosion occurred at 15,700 psi

Maximum compressive hoop stress at test termination: 230,000 psi





REPORT DOCUMENTATION PAGE

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6. AUTHOR(S) J. D. Stachiw, NCCOSC RDT&E Division; A. Pyzik, D. Carroll, A. Prunier, Dow Chemical Company; and T. Allen, Allied Technical Services					
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13. ABSTRACT (Maximum 200 words) <p>B₄C/Al ceramic composites have been found to be excellent candidates for pressure housing applications for use in deep submergence underwater vehicles. These materials have the compressive strengths of a ceramic material but a lower weight/displacement ratio and can be produced in near-net shape. A methodology has been developed in The Dow Chemical Company to form large parts of this material. Nine B₄C/Al cylinders with 6-inch diameters and lengths of up to 9 inches were produced in the first phase of the program for development of pressure housing for underwater vehicles. The pressure housing tests showed that the compressive strength of this material in the form of 6-inch diameter by 5-inch-long cylinders was in excess of 300 kpsi. The weight to displacement of cylinders equipped with titanium joint rings for 9000-psi service with safety factor of 2.2 was found to be 0.365, a 27-percent improvement over alumina cylinders with identical dimensions and safety factors. When B₄C/Al cylinders are compared to titanium alloy Ti₆Al₄Va cylinders for 9000-psi service with identical safety factor, the titanium cylinders are found to be 256 percent heavier.</p>					
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